SECOR

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INCORPORATED

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March 31, 2003

Mr. Russell Hart
Remedial Project Manager
United States Environmental Protection Agency
Region V
77 West Jackson Blvd
Mail Code SR-6J
Chicago, Illinois 60604-3590

RE: Remedial Design Work Plan Additional Project Plans: QAPP, FSP, HASP
Southeast Rockford Groundwater Contamination Superfund Site; Area 9/10

Dear Mr. Hart:

On behalf of Hamilton Sundstrand Corporation (HS), SECOR International Incorporated (SECOR) is submitting the enclosed additional Project Plans in support of the Work Plan for *Remedial Design for Area 9/10 of the Southeast Rockford Groundwater Contamination* Superfund Site in Rockford, Illinois. These plans consist of the Quality Assurance Project Plan (QAPP), Field Sampling Plan (FSP) and Health and Safety Plan (HASP). Each plan is an appendix to the Remedial Design (RD) Work Plan that was originally submitted to you dated February 26, 2003. An electronic copy of these documents has also been included on the enclosed compact disc.

Please note that the project schedules included in the QAPP and the FSP are the same as that presented in the original RD Work Plan submission. This schedule is currently under revision based on comments received from you on the RD Work Plan. The revised schedule along with the other changes based on your other comments will be forwarded to you for receipt on April 7, 2003.

We look forward to continuing to work with you on this effort. If you have any questions, please do not hesitate to call

Sincerely,
SECOR International Incorporated

David M. Curnock
Principal Scientist

Enclosure: RD Work Plan, Area 9/10 QAPP, FSP, HASP

cc: T. Turner, USEPA
S. Moyer, HS/UTC
E. Alletzhauser, UTC
T. Williams, IEPA
T. Ayers, IEPA

RDWPtrns033103

ATTACHMENT A TO QAPP
LABORATORY MDLS, RLS, AND CONTROL LIMITS

SECOR Project NO.: 13UN.02072.00.0001

March 31, 2003

STL Chicago
Method Limit Report

Project: Secor - Remedial Design SE Rockford Area 9/10
Date: 3/17/03

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Method: Leachable, Metals Analysis (ICAP) (6010L)										
Arsenic	6010B	TCLP	mg/L	0.01	0.1	80	120	20		
Barium	6010B	TCLP	mg/L	0.01	1	80	120	20		
Cadmium	6010B	TCLP	mg/L	0.002	0.05	80	120	20		
Chromium	6010B	TCLP	mg/L	0.01	0.05	80	120	20		
Lead	6010B	TCLP	mg/L	0.005	0.05	80	120	20		
Selenium	6010B	TCLP	mg/L	0.01	0.1	80	120	20		
Silver	6010B	TCLP	mg/L	0.005	0.05	80	120	20		
Method: Mercury (CVAA) (7470)										
Mercury	7470A	TCLP	ug/L		2	80	120	20		
Method: Volatile Organics (8260B)										
1,1,1,2-Tetrachloroethane	8260B	Water	ug/L	0.21	1	70	134	20		
1,1,1-Trichloroethane	8260B	Water	ug/L	0.22	1	66	129	20		
1,1,2,2-Tetrachloroethane	8260B	Water	ug/L	0.25	1	72	127	20		
1,1,2-Trichloroethane	8260B	Water	ug/L	0.33	1	69	138	20		
1,1-Dichloroethane	8260B	Water	ug/L	0.2	1	69	127	20		
1,1-Dichloroethene	8260B	Water	ug/L	0.19	1	54	127	20		
1,1-Dichloropropene	8260B	Water	ug/L	0.24	1	70	128	20		
1,2,3-Trichlorobenzene	8260B	Water	ug/L	0.24	1	75	123	20		
1,2,3-Trichloropropane	8260B	Water	ug/L	0.2	1	71	126	20		
1,2,4-Trichlorobenzene	8260B	Water	ug/L	0.23	1	77	123	20		
1,2,4-Trimethylbenzene	8260B	Water	ug/L	0.2	1	72	126	20		
1,2-Dibromo-3-chloropropane	8260B	Water	ug/L	0.46	1	66	123	20		
1,2-Dibromoethane (EDB)	8260B	Water	ug/L	0.25	1	71	135	20		
1,2-Dichlorobenzene	8260B	Water	ug/L	0.24	1	74	119	20		
1,2-Dichloroethane	8260B	Water	ug/L	0.25	1	63	133	20		
1,2-Dichloroethene (total)	8260B	Water	ug/L	0.42	1	72	121	20		
1,2-Dichloropropane	8260B	Water	ug/L	0.22	1	71	132	20		
1,3,5-Trimethylbenzene	8260B	Water	ug/L	0.2	1	69	123	20		
1,3-Dichlorobenzene	8260B	Water	ug/L	0.23	1	73	121	20		
1,3-Dichloropropane	8260B	Water	ug/L	0.23	1	71	133	20		
1,4-Dichlorobenzene	8260B	Water	ug/L	0.22	1	74	121	20		
2,2-Dichloropropane	8260B	Water	ug/L	0.2	1	56	141	20		
2-Butanone (MEK)	8260B	Water	ug/L	1.7	5	54	145	20		
2-Chlorotoluene	8260B	Water	ug/L	0.22	1	69	120	20		
2-Hexanone	8260B	Water	ug/L	1.2	5	70	144	20		
4-Chlorotoluene	8260B	Water	ug/L	0.22	1	68	120	20		

STL Chicago
Method Limit Report

Project: Secor - Remedial Design SE Rockford Area 9/10
Date: 3/17/03

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
4-Methyl-2-pentanone (MIBK)	8260B	Water	ug/L	0.92	5	66	147	20		
Acetone	8260B	Water	ug/L	1.5	5	43	150	20		
Benzene	8260B	Water	ug/L	0.2	1	74	116	20		
Bromobenzene	8260B	Water	ug/L	0.22	1	77	121	20		
Bromochloromethane	8260B	Water	ug/L	0.19	1	57	133	20		
Bromodichloromethane	8260B	Water	ug/L	0.23	1	76	129	20		
Bromoform	8260B	Water	ug/L	0.22	1	73	139	20		
Bromomethane	8260B	Water	ug/L	0.18	1	51	152	20		
Carbon disulfide	8260B	Water	ug/L	0.4	5	29	136	20		
Carbon tetrachloride	8260B	Water	ug/L	0.24	1	66	136	20		
Chlorobenzene	8260B	Water	ug/L	0.22	1	76	124	20		
Chloroethane	8260B	Water	ug/L	0.21	1	68	135	20		
Chloroform	8260B	Water	ug/L	0.23	1	74	128	20		
Chloromethane	8260B	Water	ug/L	0.16	1	56	129	20		
cis-1,2-Dichloroethene	8260B	Water	ug/L	0.21	1	78	126	20		
cis-1,3-Dichloropropene	8260B	Water	ug/L	0.22	1	75	123	20		
Dibromochloromethane	8260B	Water	ug/L	0.23	1	74	137	20		
Dibromomethane	8260B	Water	ug/L	0.26	1	66	131	20		
Dichlorodifluoromethane	8260B	Water	ug/L	0.14	1	56	136	20		
Ethylbenzene	8260B	Water	ug/L	0.2	1	74	121	20		
Hexachlorobutadiene	8260B	Water	ug/L	0.24	1	56	147	20		
Isopropylbenzene	8260B	Water	ug/L	0.21	1	67	123	20		
m&p-Xylenes	8260B	Water	ug/L	0.39	2	71	125	20		
Methylene chloride	8260B	Water	ug/L	0.19	1	52	133	20		
Methyl-tert-butyl-ether (MTBE)	8260B	Water	ug/L	0.21	1	52	156	20		
Naphthalene	8260B	Water	ug/L	0.34	1	69	125	20		
n-Butylbenzene	8260B	Water	ug/L	0.22	1	71	118	20		
n-Propylbenzene	8260B	Water	ug/L	0.25	1	67	123	20		
o-Xylene	8260B	Water	ug/L	0.21	1	72	124	20		
p-Isopropyltoluene	8260B	Water	ug/L	0.22	1	67	126	20		
sec-Butylbenzene	8260B	Water	ug/L	0.22	1	69	124	20		
Styrene	8260B	Water	ug/L	0.23	1	80	125	20		
tert-Butylbenzene	8260B	Water	ug/L	0.21	1	69	123	20		
Tetrachloroethene	8260B	Water	ug/L	0.2	1	69	128	20		
Toluene	8260B	Water	ug/L	0.21	1	71	122	20		
trans-1,2-Dichloroethene	8260B	Water	ug/L	0.21	1	64	119	20		
trans-1,3-Dichloropropene	8260B	Water	ug/L	0.24	1	76	126	20		

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Trichloroethene	8260B	Water	ug/L	0.21	1	70	120	20		
Trichlorofluoromethane	8260B	Water	ug/L	0.22	1	62	141	20		
Vinyl chloride	8260B	Water	ug/L	0.18	1	67	137	20		
Surrogate										
1,2-Dichloroethane-d4 (surr)	8260B	Water	ug/L						61	131
4-Bromofluorobenzene (surr)	8260B	Water	ug/L						73	122
Dibromofluoromethane (surr)	8260B	Water	ug/L						66	132
Toluene-d8 (surr)	8260B	Water	ug/L						78	128
Method: Volatile Organics (8260B)										
1,1,1,2-Tetrachloroethane	8260B	Solid	ug/Kg	0.73	5	83	123	20		
1,1,1-Trichloroethane	8260B	Solid	ug/Kg	0.61	5	63	133	20		
1,1,2,2-Tetrachloroethane	8260B	Solid	ug/Kg	0.64	5	68	139	20		
1,1,2-Trichloroethane	8260B	Solid	ug/Kg	0.71	5	71	143	20		
1,1-Dichloroethane	8260B	Solid	ug/Kg	0.88	5	63	133	20		
1,1-Dichloroethene	8260B	Solid	ug/Kg	1	5	51	132	20		
1,1-Dichloropropene	8260B	Solid	ug/Kg	0.8	5	78	148	20		
1,2,3-Trichlorobenzene	8260B	Solid	ug/Kg	0.99	5	75	125	20		
1,2,3-Trichloropropane	8260B	Solid	ug/Kg	1.1	5	71	129	20		
1,2,4-Trichlorobenzene	8260B	Solid	ug/Kg	0.79	5	76	127	20		
1,2,4-Trimethylbenzene	8260B	Solid	ug/Kg	0.82	5	74	133	20		
1,2-Dibromo-3-chloropropane	8260B	Solid	ug/Kg	1.1	5	59	124	20		
1,2-Dibromoethane (EDB)	8260B	Solid	ug/Kg	0.76	5	72	133	20		
1,2-Dichlorobenzene	8260B	Solid	ug/Kg	0.73	5	85	120	20		
1,2-Dichloroethane	8260B	Solid	ug/Kg	0.58	5	69	125	20		
1,2-Dichloroethene (total)	8260B	Solid	ug/Kg	1.9	5	63	144	20		
1,2-Dichloropropane	8260B	Solid	ug/Kg	0.96	5	76	132	20		
1,3,5-Trimethylbenzene	8260B	Solid	ug/Kg	0.58	5	72	128	20		
1,3-Dichlorobenzene	8260B	Solid	ug/Kg	0.91	5	83	122	20		
1,3-Dichloropropane	8260B	Solid	ug/Kg	0.93	5	78	127	20		
1,4-Dichlorobenzene	8260B	Solid	ug/Kg	0.89	5	84	121	20		
2,2-Dichloropropane	8260B	Solid	ug/Kg	1.3	5	67	134	20		
2-Butanone (MEK)	8260B	Solid	ug/Kg	4.2	5	50	150	30		
2-Chlorotoluene	8260B	Solid	ug/Kg	1	5	63	137	20		
2-Hexanone	8260B	Solid	ug/Kg	1.7	5	69	140	20		
4-Chlorotoluene	8260B	Solid	ug/Kg	0.77	5	76	123	20		
4-Methyl-2-pentanone (MIBK)	8260B	Solid	ug/Kg	3	5	68	134	20		
Acetone	8260B	Solid	ug/Kg	4.1	5	46	167	20		

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Benzene	8260B	Solid	ug/Kg	0.66	5	72	128	20		
Bromobenzene	8260B	Solid	ug/Kg	0.71	5	81	123	20		
Bromochloromethane	8260B	Solid	ug/Kg	0.99	5	68	129	20		
Bromodichloromethane	8260B	Solid	ug/Kg	0.68	5	74	128	20		
Bromoform	8260B	Solid	ug/Kg	0.91	5	78	132	20		
Bromomethane	8260B	Solid	ug/Kg	2.9	5	48	127	20		
Carbon disulfide	8260B	Solid	ug/Kg	2	5	23	138	20		
Carbon tetrachloride	8260B	Solid	ug/Kg	0.83	5	67	127	20		
Chlorobenzene	8260B	Solid	ug/Kg	0.91	5	83	125	20		
Chloroethane	8260B	Solid	ug/Kg	1.6	5	59	163	20		
Chloroform	8260B	Solid	ug/Kg	0.62	5	73	135	20		
Chloromethane	8260B	Solid	ug/Kg	0.94	5	45	141	20		
cis-1,2-Dichloroethene	8260B	Solid	ug/Kg	1.2	5	68	148	20		
cis-1,3-Dichloropropene	8260B	Solid	ug/Kg	0.79	5	80	124	20		
Dibromochloromethane	8260B	Solid	ug/Kg	0.69	5	77	127	20		
Dibromomethane	8260B	Solid	ug/Kg	0.69	5	70	130	20		
Dichlorodifluoromethane	8260B	Solid	ug/Kg	0.75	5	43	121	20		
Ethylbenzene	8260B	Solid	ug/Kg	1.1	5	79	123	20		
Hexachlorobutadiene	8260B	Solid	ug/Kg	1	5	66	127	20		
Isopropylbenzene	8260B	Solid	ug/Kg	0.75	5	77	118	20		
m&p-Xylenes	8260B	Solid	ug/Kg	2.1	10	79	123	20		
Methylene chloride	8260B	Solid	ug/Kg	1.8	5	58	143	20		
Methyl-tert-butyl-ether (MTBE)	8260B	Solid	ug/Kg	0.64	5	61	132	20		
Naphthalene	8260B	Solid	ug/Kg	1	5	65	132	20		
n-Butylbenzene	8260B	Solid	ug/Kg	0.84	5	65	138	20		
n-Propylbenzene	8260B	Solid	ug/Kg	0.86	5	77	124	20		
o-Xylene	8260B	Solid	ug/Kg	0.93	5	80	123	20		
p-Isopropyltoluene	8260B	Solid	ug/Kg	0.68	5	74	126	20		
sec-Butylbenzene	8260B	Solid	ug/Kg	0.81	5	77	128	20		
Styrene	8260B	Solid	ug/Kg	1	5	85	126	20		
tert-Butylbenzene	8260B	Solid	ug/Kg	0.78	5	79	124	20		
Tetrachloroethene	8260B	Solid	ug/Kg	0.67	5	75	129	20		
Toluene	8260B	Solid	ug/Kg	1	5	75	125	20		
trans-1,2-Dichloroethene	8260B	Solid	ug/Kg	0.94	5	58	139	20		
trans-1,3-Dichloropropene	8260B	Solid	ug/Kg	0.84	5	75	134	20		
Trichloroethene	8260B	Solid	ug/Kg	0.59	5	75	129	20		
Trichlorofluoromethane	8260B	Solid	ug/Kg	0.71	5	57	135	20		

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Vinyl chloride	8260B	Solid	ug/Kg	0.74	5	58	140	20		
Surrogate										
1,2-Dichloroethane-d4 (surr)	8260B	Solid	ug/Kg						50	145
4-Bromofluorobenzene (surr)	8260B	Solid	ug/Kg						60	140
Dibromofluoromethane (surr)	8260B	Solid	ug/Kg						60	140
Toluene-d8 (surr)	8260B	Solid	ug/Kg						66	141
Method: Volatile Organics (8260B)										
1,1,1,2-Tetrachloroethane	8260B	High/MeOH	ug/Kg	25.5	100	74	120	30		
1,1,1-Trichloroethane	8260B	High/MeOH	ug/Kg	16.5	100	69	133	30		
1,1,2,2-Tetrachloroethane	8260B	High/MeOH	ug/Kg	18.5	100	70	126	30		
1,1,2-Trichloroethane	8260B	High/MeOH	ug/Kg	31.5	100	67	133	30		
1,1-Dichloroethane	8260B	High/MeOH	ug/Kg	13.5	100	68	119	30		
1,1-Dichloroethene	8260B	High/MeOH	ug/Kg	14	100	44	143	30		
1,1-Dichloropropene	8260B	High/MeOH	ug/Kg	18.5	100	65	134	30		
1,2,3-Trichlorobenzene	8260B	High/MeOH	ug/Kg	49	100	68	117	30		
1,2,3-Trichloropropane	8260B	High/MeOH	ug/Kg	49	100	64	118	30		
1,2,4-Trichlorobenzene	8260B	High/MeOH	ug/Kg	41.5	100	61	117	30		
1,2,4-Trimethylbenzene	8260B	High/MeOH	ug/Kg	23	100	69	122	30		
1,2-Dibromo-3-chloropropane	8260B	High/MeOH	ug/Kg	22.5	100	56	102	30		
1,2-Dibromoethane (EDB)	8260B	High/MeOH	ug/Kg	25.5	100	69	122	30		
1,2-Dichlorobenzene	8260B	High/MeOH	ug/Kg	17	100	76	125	30		
1,2-Dichloroethane	8260B	High/MeOH	ug/Kg	21.5	100	64	115	30		
1,2-Dichloroethene (total)	8260B	High/MeOH	ug/Kg	29	100	60	139	30		
1,2-Dichloropropane	8260B	High/MeOH	ug/Kg	17.5	100	70	122	30		
1,3,5-Trimethylbenzene	8260B	High/MeOH	ug/Kg	19.5	100	66	125	30		
1,3-Dichlorobenzene	8260B	High/MeOH	ug/Kg	23	100	75	119	30		
1,3-Dichloropropane	8260B	High/MeOH	ug/Kg	23.5	100	71	118	30		
1,4-Dichlorobenzene	8260B	High/MeOH	ug/Kg	20.5	100	76	127	30		
2,2-Dichloropropane	8260B	High/MeOH	ug/Kg	11.5	100	41	131	30		
2-Butanone (MEK)	8260B	High/MeOH	ug/Kg	51	100	40	125	30		
2-Chlorotoluene	8260B	High/MeOH	ug/Kg	40.5	100	62	134	30		
2-Hexanone	8260B	High/MeOH	ug/Kg	52	100	50	116	30		
4-Chlorotoluene	8260B	High/MeOH	ug/Kg	23	100	66	131	30		
4-Methyl-2-pentanone (MIBK)	8260B	High/MeOH	ug/Kg	37.5	100	54	119	30		
Acetone	8260B	High/MeOH	ug/Kg	29	100	34	143	30		
Benzene	8260B	High/MeOH	ug/Kg	14	100	67	122	30		
Bromobenzene	8260B	High/MeOH	ug/Kg	27.5	100	74	133	30		

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Bromochloromethane	8260B	High/MeOH	ug/Kg	24.5	100	60	124	30		
Bromodichloromethane	8260B	High/MeOH	ug/Kg	19	100	66	128	30		
Bromoform	8260B	High/MeOH	ug/Kg	18	100	70	123	30		
Bromomethane	8260B	High/MeOH	ug/Kg	10.5	100	36	164	30		
Carbon disulfide	8260B	High/MeOH	ug/Kg	20.5	100	21	124	30		
Carbon tetrachloride	8260B	High/MeOH	ug/Kg	16.5	100	59	127	30		
Chlorobenzene	8260B	High/MeOH	ug/Kg	22	100	80	125	30		
Chloroethane	8260B	High/MeOH	ug/Kg	20	100	33	207	30		
Chloroform	8260B	High/MeOH	ug/Kg	18	100	61	129	30		
Chloromethane	8260B	High/MeOH	ug/Kg	23.5	100	55	129	30		
cis-1,2-Dichloroethene	8260B	High/MeOH	ug/Kg	17	100	64	144	30		
cis-1,3-Dichloropropene	8260B	High/MeOH	ug/Kg	22.5	100	68	123	30		
Dibromochloromethane	8260B	High/MeOH	ug/Kg	19	100	70	119	30		
Dibromomethane	8260B	High/MeOH	ug/Kg	22.5	100	67	121	30		
Dichlorodifluoromethane	8260B	High/MeOH	ug/Kg	12	100	29	135	30		
Ethylbenzene	8260B	High/MeOH	ug/Kg	22.5	100	78	128	30		
Hexachlorobutadiene	8260B	High/MeOH	ug/Kg	38.5	100	63	126	30		
Isopropylbenzene	8260B	High/MeOH	ug/Kg	20	100	67	133	30		
m&p-Xylenes	8260B	High/MeOH	ug/Kg	50	200	76	133	30		
Methylene chloride	8260B	High/MeOH	ug/Kg	20	100	57	129	30		
Methyl-tert-butyl-ether (MTBE)	8260B	High/MeOH	ug/Kg	30.5	100	47	126	30		
Naphthalene	8260B	High/MeOH	ug/Kg	38	100	51	158	30		
n-Butylbenzene	8260B	High/MeOH	ug/Kg	18.5	100	64	118	30		
n-Propylbenzene	8260B	High/MeOH	ug/Kg	27.5	100	69	130	30		
o-Xylene	8260B	High/MeOH	ug/Kg	23.5	100	74	127	30		
p-Isopropyltoluene	8260B	High/MeOH	ug/Kg	23.5	100	68	129	30		
sec-Butylbenzene	8260B	High/MeOH	ug/Kg	20.5	100	69	139	30		
Styrene	8260B	High/MeOH	ug/Kg	28.5	100	80	129	30		
tert-Butylbenzene	8260B	High/MeOH	ug/Kg	13.5	100	71	125	30		
Tetrachloroethene	8260B	High/MeOH	ug/Kg	23	100	75	125	30		
Toluene	8260B	High/MeOH	ug/Kg	18	100	72	123	30		
trans-1,2-Dichloroethene	8260B	High/MeOH	ug/Kg	13.5	100	66	138	30		
trans-1,3-Dichloropropene	8260B	High/MeOH	ug/Kg	19.5	100	60	115	30		
Trichloroethene	8260B	High/MeOH	ug/Kg	21.5	100	70	123	30		
Trichlorofluoromethane	8260B	High/MeOH	ug/Kg	19.5	100	59	145	30		
Vinyl chloride	8260B	High/MeOH	ug/Kg	18	100	61	135	30		
Surrogate										

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
1,2-Dichloroethane-d4 (surr)	8260B	High/MeOH	ug/Kg						43	139
4-Bromofluorobenzene (surr)	8260B	High/MeOH	ug/Kg						57	124
Dibromofluoromethane (surr)	8260B	High/MeOH	ug/Kg						64	132
Toluene-d8 (surr)	8260B	High/MeOH	ug/Kg						70	128
Method: Jet Fuel-4 (8015D)										
Jet Fuel #4	8015B	Water	mg/L	0.125	0.125	31	103	20		
Surrogate	8015B	Water	mg/L							
2-Fluorobiphenyl (surr)	8015B	Water	mg/L						25	129
o-Terphenyl (surr)	8015B	Water	mg/L						37	159
Method: Jet Fuel-4 (8015D)										
Jet Fuel #4	8015B	Soil	mg/kg	4.2	4.2	50	150	20		
Surrogate	8015B	Soil	mg/kg							
2-Fluorobiphenyl (surr)	8015B	Soil	mg/kg						33	115
o-Terphenyl (surr)	8015B	Soil	mg/kg						34	168

Notes:

MDLs will vary based on annual performance.

RLs will vary based on sample volume/size; dilution factors; dry weight reporting (soils) and annual MDL determinations.

Lower/Upper Control Limits (LCL/UCL) are listed for the LCS and MS/MSD for Organics; LCS limits for TCLP are listed, however, MS limits (post-extraction spikes) are 50-150%.

For Method 8260B, the laboratory will only control the analysis on the highlighted/italicized LCS compounds - not the entire compound list.

ATTACHMENT B TO QAPP
LABORATORY STANDARD OPERATING PROCEDURES

SECOR Project NO.: 13UN.02072.00.0001

March 31, 2003

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TITLE: Gas Chromatography Mass Spectrometry - Volatiles SW-846 Method 8260

Updated by:	Signature:	Date:
JoAnn Petruszak Supervisor, GC/MS Volatiles		10-22-02

Approved by:	Signature:	Date:
Gary Rynkar Section Manager, GC/MS Dept.		10/22/2
David L. Kaczka Env. Health & Safety Coor.		11/6/02
Terese Preston Quality Manager		11/6/02

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ISSUED TO : Mark Densmore, Secor
Re: UTC Proposal

Full Signature Approvals Are Kept on File
with STL's QA Standard Practice Records

1.0 SCOPE / APPLICATION

To outline the guidelines for the analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) using SW-846 Methods 8260B and 8000B as references. The preparation of all volatile samples is based on Methods 5000, 5030A and 5030B. Method 5035 is covered by a separate SOP (USP-5035), but can also be found in this SOP.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

1.1.2 Reporting Limits

Reporting Limits [a.k.a., Estimated Quantitation Limits (EQLs)] are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory maintains reporting limits higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported.

Method detection level studies are performed annually, and reporting limits are assessed. If the MDL does not meet the routine laboratory reporting limit or the method specified limit, it is repeated or the laboratory reporting limit is reassessed. If the laboratory continually demonstrates that the method reporting limits are not achieved, equipment, technique, and the method are reviewed to assure optimal performance or appropriate action is taken. Table 1 defines the reporting limits and analyte list for SW-846 Method 8260B.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM, Revision 02).

1.2 Summary of Method

This method is used to determine volatile organic compounds in a variety of matrices. It is applicable to water, soil, sediment, sludge and waste drum samples.

This method can be used to quantify most volatile organic compounds that have a boiling point less than 200°F. It is also limited to those compounds that elute as sharp peaks from a capillary column. A listing of applicable compounds and their characteristic ions appears in Table 2.

A portion of sample, measured into a sample vessel, is purged with an inert gas. The volatile compounds are transferred to a trap, containing retarding materials. The trap is then backflushed with the inert gas and rapidly heated to effectively transfer the compounds to the GC column. The GC oven is then, temperature ramped to separate the compounds and introduce them to the source. The mass filter separates the ions, which are then detected by the analyzer. The data system then provides qualitative and quantitative information concerning the sample.

Instrument calibration occurs about every 12 hours, or prior to analysis. Instrument maintenance is performed as needed or daily basis.

2.0 INTERFERENCES

1. External interferences can be caused by contaminants from sample containers, preparative glassware and reagents, syringes and columns and manifest themselves as high background and/or discrete peaks. Some contaminants are also introduced through the sample vial seal and/or instrument sample connections. Proper glassware preparation, sample handling and instrument maintenance should eliminate these sources. A laboratory method blank (MB) is analyzed prior to any analysis to show absence of any contaminants. Reagent water sampled in the lab and carried through all field operations is also analyzed to show absence of contaminants from field sampling.

2. Carryover is also another source of contamination. Any time a high level sample is analyzed, the next sample in the batch is checked for carryover. If carryover is suspected, that sample is re-analyzed. The position is rinsed with methanol/water. If the carryover is excessive and continues into the next samples, the batch is aborted/paused, the column

and trap baked, and/or blanks analyzed until all contamination is absent. If further response is required (i.e., trap replacement), it is documented in the maintenance logbook. Refer to Section 7.4 for information on preventive maintenance.

3. Internal interferences can be purged from the sample with the target compounds and appear as elevated baselines or distinct peaks. Internal interferences most often manifest themselves as low/high recoveries of surrogate/matrix spike compounds. Matrix interferences vary from sample to sample.

4. The volatile lab must be free of solvents. All analytes must be less than their EQL (Estimated Quantitation Limit). The volatile lab is under positive pressure to reduce lab contamination, however, intermittent low levels of acetone and methylene chloride may be detected, usually below the EQL. Refer to Section 8.2 (Corrective Action) for clarification for blank contamination.

3.0 SAFETY

- All employees will adhere to the practices and policies in the STL Corporate Safety Manual (CSM) and will read the MSDS's for the materials used in this method before handling or using the material.
- Special care needs to be taken with the solvents used in this method.
- Interior parts of the GC/MS can be very hot. Care should be taken during maintenance.

4.0 EQUIPMENT AND SUPPLIES

4.1 Current Hardware/Software

- 3 Hewlett-Packard 5890 GC interfaced with a 5971 MSD. Equipped with DB-624 column.
- 3 Hewlett-Packard 5890 GC interfaced with a 5972 MSD. Equipped with DB-624 column.
- 1 Hewlett-Packard 6890 GC interfaced with a 5973 MSD. Equipped with DB-624 column.
- 6 Tekmar 3000 concentrators, 1 PTS Enchon concentrator in connection with 2 Tekmar 2016 Autosamplers for two systems and 5 Varian Archon Autosamplers for four systems.
- 1 Combi PAL Static Headspace Screener in connection with Hewlett-Packard 5890 GC interfaced with a FID equipped with DB-624 column.
- 8-Hewlett-Packard Chemstations B.02.04 software and peripheral hardware.
- 1-Hewlett-Packard Chemserver 9000 series running HP-UX10.2 OS with Target 3.5.

The GC/MS has a temperature programmable chromatograph interfaced with a mass-selective detector capable of scanning from 35 - 260 amu every second or less using 70 volts of electron energy in the electron ionization mode. The system is capable of producing an acceptable spectrum of bromofluorobenzene when 50 ng/5 mLs is purged.

4.2 Data System

The analytical systems are interfaced with stand alone PC's which are Pentium based systems running Agilent Chemstation. This system is capable of continuous acquisition and storage of mass spectral data. Completed data files are automatically transferred to the Chemserver Target 3.5 processing software which is capable of plotting specific masses versus time or scan numbers (Extracted Ion Current Profile) and integration of that abundance. The system also stores the data. The NBS Library resides on the Chemserver.

4.3 Data File Name/ Batch Directory Assignment

Each job # is assigned a code at the time that the first sample is analyzed. Tune, standard, blank, and laboratory control sample (LCS) data files are designated by specific letters unique to each instrument in conjunction with the appropriate month and day (example : 3a0318 = instrument #3, first 12 hour BFB tune, March 18). During transfer of the files to the Chemserver, a unique batch directory is created on Target per instrument, date and tune.

4.4 Miscellaneous

- assorted syringes (10, 25, 50, 100, 500 and 1000 uL)
- 5 mL luer-lock gas-tight syringes
- assorted purge vessels (water, 5/25 mL)
- top-loading balance, capable of weighing to ± 0.1 g, stainless steel spatula
- assorted amber and clear Teflon-lined screw-capped vials (1.5-2.0 mL, 3.5-5.0 mL)
- cleaned 40 mL vials w/Teflon-lined screw-caps
- assorted volumetrics (10 mL, 25 mL, 50 mL and 100 mL)

5.0 REAGENTS AND STANDARDS

The majority of the calibration standards are EPA certified, A2LA or second-source verified by the standard vendor in situations where suitable SRMs (Standard Reference Material) was available. For those compounds where standards must be made from neat material (due to instability) or some non-routine compounds, **where available**, a second-source is purchased and used in the LCS to verify the standard

Each time new standards are prepared and a new initial calibration is required, the standards are verified against a second-source LCS prior to any sample analysis. This holds for all routine compounds and those available as second-source material in the LCS.

All neat standards/kits received are entered into LabNet (LIMS) (and recorded in the Neat Standards Logbook). A code is written on the bottle/kit and entered into LabNet (and recorded in the logbook). All neat standards are then stored in a separate freezer at approximately –10 °C until needed. The standard is issued a unique ID# [i.e., Neat Standards Reference Number (NSRN)] which is used to track all standards as they are used as is or in preparation of stock/working solutions. The format of the standards in LabNet will prevent working or intermediate level solutions from being used past the expiration date of the neat or stock solutions.

5.1.1 Reagent Water

One (1) liter of water is continuously purged with pre-purified nitrogen. The reagent water is routinely demonstrated to be interference-free. All compounds are < EQL or 5x EQL for methylene chloride and acetone.

5.1.2 Methanol

Methanol is purchased from B&J (Purge and Trap interference-free). Each lot number of methanol is checked for contamination prior to laboratory use and is documented in the Methanol Lot Number Logbook in the GC/MS VOA department.

5.2 Surrogate Spiking Solution

Stock surrogates are purchased as a mix from Ultra. The following surrogates are used:

Compound	Concentration
4-Bromofluorobenzene	\
1,2-Dichloroethane-d_4	2500 ppm
Toluene-d_8	/
Dibromofluoromethane	

The transfer is entered into LabNet (and recorded in the Standard Preparation Log). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier). Working surrogate solution is prepared at the same time as the internal standard solution (Section 5.3.)

- Life of Standard: 1-year unopened; once opened, they are used for a period of 6 months or until used.
- Storage Requirements: Stored in a freezer at approximately –10 °C in the dark and kept for a period of one year unopened. *

* If the stock solution has manufacturers' expiration date, that is assigned. If the date is not evident, one year is assigned to un-opened ampules. This is applicable for all "neat" standards.

5.3 Internal Standard Spiking Solutions

Stock internal standards are purchased as a neat solution from Ultra in 1.5 -2.0 mL ampules. The following internal standards are used:

Compound	Concentration
Pentafluorobenzene	\
Chlorobenzene-d_5	2000 ppm
1,4-Difluorobenzene	/
1,4-Dichlorobenzene-d_4	

After opening, the remaining mixture is transferred to a 1.5 - 2.0 mL amber Teflon-lined screw-capped vial. The transfer is entered into LabNet (and recorded in the Standard Preparation Log). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: 1-year unopened; once opened, they are used for a period of 6 months or until used.
- Storage Requirements: Stored in a freezer at approximately –10 °C in the dark and kept for a period of one year unopened. *

* If the stock solution has manufacturers' expiration date, that is assigned. If the date is not evident, one year is assigned to un-opened ampules. This is applicable for all "neat" standards.

Compound	Volume (uLs)	MeOH (mLs)	Concentration
Internal Standard			
Pentafluorobenzene	\	\ diluted to	\
Chlorobenzene-d_5	625	25 mLs	50 ppm
1,4-Difluorobenzene	/	/	/
1,4-Dichlorobenzene-d_4			
Surrogate			
Bromofluorobenzene	\	\ diluted to	\
1,2-Dichloroethane-d_4	500	25 mLs	50 ppm
Toluene-d_8	/	/	/
Dibromofluoromethane			

NOTE: All standard 'recipes' are listed here in this SOP for guidelines for standard preparation. These 'recipes' are subject to change.

All standard preparation is entered into LabNet (and recorded in the Standards Preparation Logbook). All standard labels contain the following information: standard description, concentration, date prepared, analyst, and expiration date. Addition of 5 uL of each solution to 25 mLs of sample results in a concentration of 10 ppb per each component.

- Life of Standard: Working solutions have an expiration date of 2 weeks.
- Storage Requirements: These are stored in 1.5 - 2.0 mL amber Teflon-lined screw-capped vials at approximately –10°C in the dark.

Stock Purgeables
Calibration Standards

VOC Mix (No Gases)
2000 ug/mL in Methanol

1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethylene
1,1-Dichloropropylene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3,5-Trimethylbenzene
1,3-Dichlorobenzene
1,3-Dichloropropane
1,4-Dichlorobenzene
2,2-Dichloropropane
2-Chlorotoluene
4-Chlorotoluene
4-Isopropyltoluene
Benzene
Bromobenzene
Bromochloromethane
Bromoform
Carbon tetrachloride
Chlorobenzene
Chlorodibromomethane
Chloroform
cis-1,2-Dichloroethylene
cis-1,3-Dichloropropylene
Dibromomethane
Dichlorobromomethane
Dichlorobromomethane
Dichloromethane
Ethylbenzene
Hexachlorobutadiene
Isopropylbenzene
m-Xylene
n-Butylbenzene
n-Propylbenzene
Naphthalene
o-Xylene, p-Xylene
sec-Butylbenzene
Styrene
tert-Butylbenzene
Tetrachloroethylene
Toluene
trans-1,2-Dichloroethylene
trans-1,3-Dichloropropylene
Trichloroethylene

ICAL 2 STD Custom Mix
2000 ug/ml in Methanol
2-Methylnaphthalene
1,3,5-Trichlorobenzene
1,3-Butadiene
Isopropylether
Methyl Acetate
Hexane
Heptane
Cyclohexane
Ethyl ether
Methyl Cyclohexane

APIX Custom STD
2000, 8000 & 10000 ug/mL
Allyl Chloride
Ethyl Methacrylate
Methyl Methacrylate
Methacrylonitrile
Pentachloroethane
Trans-1,4-Dichloro-2-Butene
Iodomethane
Isobutanol
Cyclohexanone
n-Butanol
2-Nitropropane
Ethyl Acetate

Chloroprene
5000 ug/mL

SS Volatile Organic Compound Mix
2000 ug/mL in Methanol
Chloroethane
Methyl Bromide (Bromomethane)
Methyl chloride (Chloromethane)
Trichlorofluoromethane
Dichlorodifluoromethane
Vinyl chloride

Vinyl Acetate
2000 ug/mL in Methanol

Trichlorotrifluoroethane
2000 ug/mL in methanol

Volatile Ketone
Acetone
2-Hexanone
Methyl ethyl ketone
4- Methyl-2-pentanone
5000 ug/mL in Methanol

Carbon Disulfide
2000 ug/mL in Methanol

MTBE
2000 ug/mL in Methanol

THF
2000 ug/mL in methanol

Chlorohexane
1000 ug/mL in methanol

Nitriles and Acrolein Mix
Acetonitrile
Acrylonitrile
Propionitrile
Acrolein

2-Chloroethylvinylether
2000 ug/mL in methanol

5.4 Stock Purgeable Standards

These are obtained as neat solutions from Ultra, Supelco and Restek. The contents of each solution and concentration appear on the previous page. Upon opening, all contents are transferred to 1.5 - 2.0 mL amber, Teflon-lined screw-capped vials. Listed are compounds in the EPA TCL and includes compounds done on a regular basis. Other standards, if needed, are either purchased as neat solutions or neat standards from Supelco, Chem Service or other certified supplier. See appropriate entries in LabNet.

5.4.1.1 Main 8260 Mix

Waters

Stock Compound/Mix	Volume (uLs)	Vol. (MeOH)	Conc.
2000 ppm VOC MegaMix™	100	Diluted to	100 ppm
2000 ppm Trichlorotrifluoroethane	100	2 mLs	each component

5.4.1.2 Gases

Waters

Mix	Volume (uLs)	MeOH (mLs)	Conc.
2000 ppm Gas Mix	100	Diluted to 2 mL	100 ppm each component

5.4.1.3 Extra Compounds

Waters

Stock Compound/Mix	Volume (uLs)	MeOH (mLs)	Conc.
5000 ppm VOA CAL Mix 1	40	Diluted	100 ppm each component
2000 ppm CEVE	100	to	
2000 ppm Carbon Disulfide	100	2 mL	
2000 ppm Vinyl Acetate	100		

2000 ppm Tetrahydrofuran	500	Diluted	1000 ppm THF
2000 ppm MTBE	50	to	100 ppm MTBE
1000 ppm Chlorohexane	100	1 mL	100 ppm Chlorohexane

The Acrolein/Nitriles Working Standard is a vial transfer:

Compound / TCL Mix	Volume[1] (mL)	Concentration (ppm)
Nitriles/Acrolein Custom Mix Stock Std	1	800 ppm Nitriles 4000 ppm Acrolein

5.4.1.4 Main 8260 Mix

Soils

Stock Compound/Mix	Volume (uLs)	MeOH (mLs)	Conc.
2000 ppm VOC MegaMix™ 2000 ppm Trichlorotrifluoroethane	100 100	Diluted to 2 mL	100 ppm each component

5.4.1.5 Gases

Soils

Mix	Volume (uLs)	MeOH (mLs)	Conc.
2000 ppm Gas Mix	100	Diluted to 2 mL	100 ppm each component

5.4.1.6 Extra Compounds

Soils

Stock Compound/Mix[1]	Volume (uLs)	MeOH (mLs)	Conc.
5000 ppm VOA CAL Mix 1 2000 ppm CEVE 2000 ppm Carbon Disulfide 2000 ppm Vinyl Acetate	40 100 100 100	Diluted to 2 mL	100 ppm each component
2000 ppm Tetrahydrofuran 2000 ppm MTBE 1000 ppm Chlorohexane	50 50 100	Diluted to 1 mL	100 ppm each component

The Acrolein/Nitriles Working Standard is a vial transfer:

Compound/TCL Mix	Volume[1] (mL)	Concentration (ppm)
Nitriles/Acrolein Custom Mix Stock Std	1	800 ppm Nitriles 4000 ppm Acrolein

- Life of Standard: Working solutions have an expiration date of 2-weeks/1 week respectively.
- Storage Requirements: These mixtures are stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at approximately –10 °C in the dark

5.4.1.7 Appendix IX (either matrix)

Mix	Volume (uLs)	MeOH (mLs)	Conc.
2000 ppm Appendix IX	250	Diluted to 2 mLs	250 ppm Appendix IX
5000 ppm Iodomethane	100		250 ppm Iodomethane
20,000 ppm Isobutanol	1000		10,000 ppm Isobutanol

5.4.1.8 Low Level Standard

A low level standard is prepared by making a 1/10 dilution of the stock standards of each of the above (nitriles and acrolein included). This standard is used to prepare the low points in the initial calibration. The low level standard may contain the Main 8260 Mix, gases, nitriles and acrolein, and any other required standard. A low-level standard for the Appendix IX compounds is also prepared separately due to duplication of some compounds.

A low level surrogate solutions is also prepared by a 1/10 dilution of the working for low points in the water curve. The calibration levels may vary with the compounds. See recipes in the calibration section for the levels. The low point in the calibrations is based on each compounds reporting limit.

All solutions are stored in a 1.5 - 2.0 mLs amber Teflon-lined screw-capped vials at –10 °C in the dark. All standard preparation is recorded in the LabNet system. Solutions are prepared every two weeks (1 week for the gases; 1 month for the nitriles).

Purgeable Spike Standard Mixes
ACCUSTANDARD and Absolute Standard

VOC LIQUID MIXTURE
2000 ug/mL in Methanol

1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethylene
1,1-Dichloropropylene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3,5-Trimethylbenzene
1,3-Dichlorobenzene
1,3-Dichloropropane
1,4-Dichlorobenzene
2,2-Dichloropropane
2-Chlorotoluene
4-Chlorotoluene
4-Isopropyltoluene
Benzene
Bromobenzene
Bromoform
Carbon tetrachloride
Chlorobenzene
Chlorodibromomethane
Chloroform
cis-1,2-Dichloroethylene
cis-1,3-Dichloropropylene
Dibromomethane
Dichlorobromomethane
Dichloromethane
Ethylbenzene
Hexachlorobutadiene
Isopropylbenzene
m-Xylene
n-Butylbenzene
n-Propylbenzene
Naphthalene
o-Xylene
p-Xylene
sec-Butylbenzene
Styrene
tert-Butylbenzene
Tetrachloroethylene
Toluene
trans-1,2-Dichloroethylene
trans-1,3-Dichloropropylene
Trichloroethylene

Bromochloromethane
2000 ug/mL in methanol

Heptane
2000 ug/mL in methanol

MTBE
2000 ug/mL in Methanol

1,3,5-Trichlorobenzene
2000 ug/mL in methanol

Clorohexane
1000 ug/mL in methanol

Ethyl Ether
1000 ug/mL in methanol

Hexane
1000 ug/mL in methanol

Trichlorotrifluoroethane
2000 ug/mL in methanol

Volatile Organic Compound Gas Spike
2000 ug/mL in Methanol
Chloroethane
Methyl Bromide (Bromomethane)
Methyl chloride (Chloromethane)
Trichlorofluoromethane
Dichlorodifluoromethane
Vinyl chloride

Volatile Mix Additional Spike Compounds
2000 ug/mL in Methanol
Acetone
2-Hexanone
Methyl ethyl ketone
4- Methyl-2-pentanone
Carbon Disulfide
Vinyl Acetate
2-Chloroethylvinylether
Iodomethane

THF
2000 ug/mL in methanol

5.5 Stock Matrix Spike Solution

The matrix spike compounds are obtained as solutions from a second source (i.e., Accustandard) in 1.5-2.0 mL ampules. These are listed on the previous page. A different analyst than the one who prepared the calibration solutions usually prepares matrix spike solutions. These are stored at approximately -10°C in the dark prior to use. Neat standards are kept for a period of one year un-opened or the manufacturer's expiration date. Once opened, the stock may be used for 3 months.

The matrix spike solutions are prepared as follows:

5.5.1 VOC Spike

Stock Compound/Mix	Volume (uLs)	MeOH (mLs)	Conc.
2000 ppm VOC Liquid Spike	25	Diluted to 1 mL	50 ppm each component
2000 ppm 8260 Additional Spike	25		
1000 ppm 1,3,5-Trichlorobenzene Spike	50		

5.5.2 Gas Spike

Mix	Volume (uLs)	MeOH (mLs)	Conc.
2000 ppm Gas Spike	50	Diluted to 2 mL	50 ppm each component

5.5.3 Additional Spike Compound Mix

Compound	Volume[1] (uLs)	Volume (MeOH)	Concentration
2000 ppm Bromochloromethane	25	Diluted to 1 mL	50 ppm each component
1000 ppm Ethyl Ether	50		
1000 ppm Chlorohexane	50		
1000 ppm Hexane	50		
2000 ppm MTBE	25		
1000 ppm Heptane	50		
1000 ppm Trichlorotrifluoroethane	50		

5.5.4 Tetrahydrafuran Spike

Mix	Volume (uLs)	MeOH (mLs)	Conc.
2000 ppm Tetrahydrafuran	50	Diluted to 1 mL	100 ppm

For waters, addition of 5 uLs of each solution results in all spike compounds at 10 ppb, with the exception of THF which is at 20 ppb.

For soils, addition of 5 uLs of each solution, except THF at addition of 2.5 uLs, results in all compounds at 50 ppb.

These solutions are stored at approximately -10°C in several 1.5 -2.0 mL amber Teflon-lined screw-capped vials. All standard preparation is recorded in the LabNet system. Working matrix spike solutions have a 2-week/1-week expiration date or until low recoveries of the matrix spike compounds indicate a new solution is needed. See above for label information.

5.6 Stock BFB Solution

The BFB standard is purchased as a neat solution from Supelco.

Stock	Amount	MeOH (mL)	Concentration
2000 ppm BFB	25 uLs	diluted to 2 mLs	25 ppm

- Life of Standard: This stock can be kept for a period of one year until opening. Upon opening, the solution is transferred to a 1.5 - 2.0 mL vial and assigned an SRN. Once opened, it is used for a period of 6 months.
- Storage Requirements: The standard is stored at approximately –10°C in the dark

Addition of 2 uLs to 5 mLs results in a concentration of 50 ng/5 mLs.
All preparation is recorded in the LabNet system. All labels are completed as above.

NOTE: Intermediate and Working Solutions are never assigned an expiration date exceeding the expiration date of the neat/stock standards/solutions.

5.6 Reagents

5.6.1 Reagent Water

One (1) liter of water is continuously purged with pre-purified nitrogen. The reagent water is routinely demonstrated to be interference-free. All compounds are less than their EQL.

5.6.2 Methanol

Methanol is purchased from B&J (Purge and Trap interference-free). Each lot number of methanol is checked for contamination prior to laboratory use and is documented in the Methanol Lct Number Logbook in the GC/MS VOA department.

6.0 CALIBRATION

Before an instrument is used as a measuring device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration is documented.

6.1 PFTBA Autotune or Manual Tune

The instrument is first tuned in one of two ways: autotune or manual tune. The ion abundances in the calibration gas are best monitored near the temperature of analysis of BFB. Monitoring at this temperature produces the most representative cal gas scan and therefore the best estimate of BFB response.

1. If an AUTOTUNE is to be done, continue below. If not, skip to step 6. An autotune is not run before every initial calibration. If the instrument has been down for any reason previously listed or major difficulties in manual tune are encountered, an autotune is performed. Autotunes are generally NOT performed when an existing initial calibration is being met.

2. The Enviroquant software has a menu driven tune program. Begin the autotune program. Key masses are 69, 219 and 502.

3. Follow instructions and retrieve a hardcopy of the autotune results. Check the following:

- passed/fail: in itself, not necessarily an indication of MS performance
- repeller and ion focus settings
- electron multiplier voltage

4. The repeller and EM voltages are good indicators of the sources' cleanliness. Generally, the lower the setting the cleaner the source. Other factors may however, supersede (i.e., the age of the multiplier) and a clean source will not always autotune these low. The EM is set by autotune program to produce a target abundance for mass 69 (varies depending on the tune program and instrument). The operator may plan on having to increase this by 100-200 to achieve normal analysis sensitivity (depends on the tune program and the instrument).

5. Observe peak shape, absence of lead-ons/tailing, the resolution between isotopes, peak width and mass axis. A hardcopy of the profile scan is desirable, and can be filed with the autotune results.

6. If an AUTOTUNE has just been performed, continue here. If not, skip to step 9. Enter MANUAL TUNE and read the autotune (which was automatically stored in a file). For volatiles, edit the scan parameters to monitor ions 69, 131 and 219.

7. Enter one of several methods available and adjust the parameters (usually the ion focus, entrance lens and amu gain) to achieve the following relative abundances:

Mass	Relative Abundance
69	100%
131	32-40%
219	35-45%

These will vary with the MS. Mass 219 is usually 5-9% greater than mass 131. If necessary, adjust the amu gain for peak shape and high-end isotope resolution. An overall peak-width of 0.500 is desirable.

Again, these adjustments and relative abundances may not guarantee that BFB will meet requirements, but is a good place to start.

8. Hardcopy the profile scan. This should be filed with the autotune results. This file can serve as a diagnostic tool and can also provide a starting point in the event the operator has trouble meeting the initial calibration.

Save the changes to the appropriate Tune File. Exit the program.

9. If an AUTOTUNE has not been performed, enter MANUAL TUNE and adjust any parameters, if need be. Adjustment may not be necessary, and not desirable, if problems in tuning or meeting the initial calibration have not been encountered. Hardcopy a profile scan and exit.

6.2 BFB Analysis

Once the instrument is tuned, a 50 ng/5 mLs injection of 4-Bromofluorobenzene must meet criteria. The BFB can be purged or directly injected. The mass spectrum must meet the following criteria:

Mass	Ion Abundance
50	15-40% of mass 95
75	30-60% of mass 95
95	Base Peak, 100% rel. abund.
96	5 - 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95

Mass	Ion Abundance
175	5 - 9% of mass 174
176	>95% but <101% of mass 174
177	5 - 9% of mass 176

The BFB is analyzed by one of the methods in Attachment 1. **(Method parameters listed in the appendices are examples only. This statement applies to all references made to these methods).** Typical Tekmar conditions also appear in Attachment 1. The EM voltage may be 100-200 volts above autotune. The abundances of the designated masses above MUST meet the criteria before analyses can begin. If necessary, enter MANUAL TUNE and adjust parameters. The instrument is tuned about every 12 hours of analysis.

6.3 Description of Initial Calibration

An initial calibration may be completed:

- as needed - continuing calibration can not be met
- after a source cleaning and/or column change or any time a major repair or change has occurred with the instrument that affects calibration where a new calibration is indicated.

Confirm that the GC/MSD or bench-top is stable and equilibrated. If at all possible, allow the instrument to equilibrate overnight at all operating temperatures if the source/column has been cleaned/changed. Prior to beginning initial calibration it is a good idea to:

- check the background of air/water levels and base ion by scanning for appropriate ions and also visually inspecting the spectrum scan for any other possible and undesirable background.
- recheck the multiplier settings, after a source is cleaned the EM can most often be dropped.

6.4 Initial Calibration

Each calibration standard is analyzed according to one of the methods in Attachment 1. These are examples. The actual number of points in the calibration is determined by the calibration and acceptance criteria table (Attachment 4). The EM voltage may be 100-200 volts above autotune.

Allow standards to come to ambient temperature.

Fill ten 5 mLs or 25 mLs (must be loaded separately) luer-lock gas-tight syringes with reagent water to overflowing. Replace the plunger and invert. Adjust to 5 mLs (or 25 mLs), confirming the absence of any air bubbles. Pull back slightly on the plunger to allow addition of standards. Use the following as guides :

Waters: 25-mL Purge Volume

Conc. Level[1]						Recipe (uLs)					
Main Mix /Gas/ MTBE/ Other	Extra	THF	Nit	Acrol	APIX	Main Mix/Gas/ MTBE/THF/ Other (100 ppm)	LL Extra 5 ppm	Surr. (50 ppm)	Nit/Acrol (800/4000 ppm)	APIX (250 ppm)	ICAL 2 (100/ 1000 ppm)
0.3	NA	NA	NA	NA	NA	1.5 (LL)	NA	1.5 (LL)	1.5 (LL)	NA	NA
0.5	NA	5	16	80	NA	2.5 (LL)	NA	2.5 (LL)	5 (LL)	1(LL)	2.5 (LL)
1	2	10	32	160	2	5 (LL)	5	5 (LL)	1	2(LL)	5 (LL)
2	4	20	48	240	5	10 (LL)	10	10 (LL)	1.5	5(LL)	10 (LL)
5	5	50	80	400	10	25 (LL)	NA	2.5	2.5	1	25 (LL)
8	8	80	112	560	15	2	NA	4	3.5	1.5	2
10	10	100	160	800	20	2.5	NA	5	5	2	2.5
14	14	140	192	960	30	3.5	NA	7	6	3	3.5
20	20	200	240	1200	40	5	NA	10	7.5	4	5
40	40	400	320	1600	60	10	NA	20	10	6	10

[1] Stocks referred to here are listed on pages 7 thru 12, and include the regular compounds, Gas, Extra compounds, Appendix IX, Nitriles and Acrolein. Appendix IX and ICAL 2 curve is separate.

Soils: 5-mL Purge Volume

Conc. Level[1]						Recipe (uLs)				
Main Mix/Gas/ MTBE/Other	Extra	THF	Nit	Acrol	APIX	Main Mix/Gas/ MTBE/THF/ Extra/Other (100 ppm)	Surr. (50 ppm)	Nit/Acrol (800/4000 ppm)	APIX (250 ppm)	ICAL 2 100/1000 ppm
2	2	2	NA	NA	NA	2 (LL)	2 (LL)	1 (LL)	1 (LL)	2 (LL)
5	5	5	40	200	10	5 (LL)	5 (LL)	2.5 (LL)	2 (LL)	5 (LL)
20	20	20	160	800	40	20 (LL)	20 (LL)	10 (LL)	8 (LL)	20 (LL)
30	30	30	240	1200	50	30 (LL)	30 (LL)	15 (LL)	10 (LL)	30 (LL)
50	50	50	400	2000	100	2.5	5	2.5	2	2.5
70	70	70	560	2800	150	3.5	7	3.5	3	3.5
100	100	100	800	4000	200	5	10	5	4	5
150	150	150	1200	6000	300	7.5	15	7.5	6	7.5
200	200	200	1600	8000	400	10	20	10	8	10

[1] Stocks referred to here are listed on pages 7 thru 12, and include the regular compounds, Gas, Extra compounds, Appendix IX, Nitriles and Acrolein. The Appendix IX curve must be analyzed separately.

The same tables appear in the standard section. Immediately add the standards to a clean and baked purge vessel. Following the parameters in Table 1, analyze the 50 ppb standard (soil samples) or the 10 ppb standard (water samples). A normal standard will appear very similar to the ones in Figures 1 and 2. Quantitate the standard against the appropriate method file. A short list example of one file appears in Attachment 2. Sufficient areas for the first internal standard will vary somewhat between instruments. Acceptable areas should be based on maintaining sufficient sensitivity for poor responders without saturating the detector at the upper end of the calibration range. Too low an area will almost guarantee poor/unsatisfactory responses of low-response compounds and too high an area will result in saturation of some compounds at higher levels, resulting in false low response factors at high concentrations.

It is helpful to analyze a medium level standard first and assess the areas before continuing with the low/high level standards.

Response factors are calculated by the data system as follows:

$$RF = \frac{A_x \times Q_s}{A_s \times Q_x}$$

Where:
A_x = ion abundance for analyte
A_s = ion abundance for its internal standard
Q_s = concentration of its internal standard
Q_x = concentration of analyte
(Response Factors have no units)

The appropriate quant ion must be in the method file. See an example of a file in Attachment 2. A listing of the target compounds with their appropriate internal standards also appears in Attachment 2. Confirm the presence of all targets and the separation of non- co-eluting compounds. Note the response factors for the gasses. If necessary, prepare new standards.

If adjustments to the acquisition parameters are necessary, make them and re-analyze the 50 ppb standard (soil samples) or the 10 ppb standard (water samples).

When a standard is analyzed and processed on target as part of the initial calibration the RF's are automatically updated in the daily method. After all initial calibration standards are processed, checked and confirmed as being accurate and passing method criteria, the initial calibration is saved to the source method. This ensures that the correct initial calibration is used for each ensuing continuing calibration check. A hardcopy of the calibration report is generated. All method criteria are assessed for compliance. Confirm that 1) all CCC's are below 30% and 2) the RF's for all SPCC compounds are >0.300 (Minimum RF for Chloromethane, Bromoform and 1,1-Dichloroethane is 0.100).

Calibration curves are evaluated following the "Evaluation and Acceptance Criteria" table (Attachment 4). For all compounds in the initial calibration with a %RSD > 15.0%, calibration curves of area ratio versus concentration using a first or higher order regression curve of the calibration curve points will be performed.

Method 8000B/8260B specifies a minimum coefficient of determination of 0.990. The methods also specify a minimum of 5 calibration points for a linear model and a minimum of 6 calibration points for a higher order regression. The laboratory, in order to meet AFCEE requirements, will analyze a minimum number of points to satisfy both the new SW846 and AFCEE. All efforts will be made to meet the minimum COD of 0.990. However, there are some compounds that historically present a problem meeting this requirement. These compounds are usually those listed in the analyte table of Method 8260B with qualifying remarks. Many of these have various known (or unknown) issues that would effect reproducibility (i.e., Acetone qualifier pp = poor purger). These typically include many of the Appendix IX compounds as well. The laboratory will take minimal action for these compounds.

The 'recipes' noted above will be modified to include the necessary calibration levels. Recipes are for guidance only and may change as needed.

An example of an acceptable initial calibration appears in Attachment 2. The BFB tune, and all standard raw data are kept near the instrument if current, otherwise can be found in a file. Each instrument has its own initial calibration.

NOTE: The actual number of points in the calibration and the low point in the calibration may vary with client and project need. Clients may have additional requirements, which would be covered in a client-specific QAP.

6.5 Daily or Continuing Calibration

Continuing calibration occurs prior to analysis.

If time remains after the initial calibration, and the 50 ppb standard (soil samples) or the 10 ppb standard (water samples) meets continuing calibration criteria, samples can be analyzed up to the 12 hour tune limit. The samples are quantitated against the average RF or appropriate as per method. See later sections describing calculations.

After having satisfied BFB tune requirements, a continuing calibration standard must be analyzed. Analyze a 50 ppb (soil samples) or 10 ppb (water samples) standard following the procedure outlined above. Confirm Form 7 that all CCC's are <20% Drift and the RF's for all SPCC's are >0.300 (Minimum RF for Chloromethane, Bromoform and 1,1-Dichloroethane is 0.100). If so, the continuing calibration is acceptable and analysis can begin.

If continuing calibration can not be met, either new standards and/or a new calibration are needed.

NOTE: Method 8260B stipulate that if the CCC's are not part of the analyte list then all compounds being reported must be < 20% drift.

All internal standard areas and retention times are assessed immediately after calibration. Areas and times compared to the mid point of the initial calibration. Internal standard areas should not deviate by a factor of two or the retention times should not deviate by > 30 s. If the situation occurs, appropriate action is taken and the standard re-analyzed. All corrective action and return to control are documented in the CAR logbook for the appropriate instrument.

7.0 PROCEDURE

7.1 Quality Control Checks

Quality Control is accomplished through 1) daily tuning and calibration checks and 2) preparation QC traceable through individual batches.

7.1.1 Initial Calibration

PFTBA		
BFB TUNE	Prior to Initial Cal	*limits in Section 6.2
200 or 40 ng \		
150 or 20 ng |		
100 or 14 ng |		
70 or 10 ng |	Initial Cal need dependent on situation.	*limits in Section 6.4
50 or 8 ng |		
30 or 5ng |		
20 or 2 ng |		
5 or 1 ng |		
0.5 ng /		

NOTE: As stated, the actual number of points in the calibration and the low point in the calibration may vary with client and project need. Minimum number of points for AFCEE and/or 3rd Edition SW-846 may be 9 (nine) or 10 (ten) depending on matrix. Other clients may have additional requirements, which would be covered in a client-specific QAP.

7.1.2 Method Blank (MB)

Prior to any analysis, the reagent water must be shown to be free of interference's and target compounds.

A 5 mL or 25 mL portion of reagent water is analyzed using one of the methods in Attachment 1. All target compounds must be less than the quantitation limit (See Section 2.0). Once the MB analysis is complete and acceptable, analysis can proceed.

7.1.3 Daily Analysis

PFTBA		
BFB	Prior to continuing	* See above calibration
Daily Calibration Standard	Prior to samples	* See Section 6.5
Samples *		

*Any given 12 hour period contains a tune, standard, blank and LCS. Preparation QC is at a 5% frequency. Instrumental controls are outlined above and further discussed in the procedure section.

Prep QC	Frequency
MB	Prior to analysis
LCS	1 set per analysis batch (see below *)
MS/MSD's[1]	at least 1 set in 20
Surrogates	every blank, sample and QC Sample
QC Charting	LCS/LCD[2] set per frequency to satisfy charting requirements.

[1] The sample selection for MS/MSD is rotated among client samples so that various matrix problems may be noted and/or addressed.
[2] LCS Duplicate (LCD) is performed when insufficient sample is available for an MS/MS.

7.2 Sample Preservation and Storage

Sample containers, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests. Listed below are the holding times and the references that include container and preservation requirements for compliance with the Resource Conservation and Recovery Act (RCRA).

Matrix	SW-846
All	14 days

All samples received for volatile analysis are refrigerated upon receipt at 4 ± 2°C. Refrigeration is the only preservative for 5030 soil samples, while water samples are additionally preserved with 3 drops of 36% HCl to a pH >2. Water samples marked as un-preserved are analyzed within 7 days.

7.3 Sample Preparation / Analysis

7.3.1 Waters

1. Allow samples and standards to come to ambient temperature.

2. Remove the plunger from a 25 mL luer-lock gas-tight syringe and fill to near over-flowing. Replace the plunger. The pH of all samples is verified at time of analysis. If the pH < 2, a check-mark is placed in the appropriate column in the logbook. If the pH > 2, the actual estimated pH is written in the same column. pH checks and verification of hold-times are documented on the review form. Samples lacking preservation may be noted in the case narrative. Invert the syringe, and adjust the volume to 25 mLs. Confirm the absence of all air bubbles.

3. Draw back slightly on the plunger. Add 5 uLs of the working ISS/SSS solutions. Immediately add the sample to a clean purge vessel. Using the methods described in Attachment 1 analyze the sample.

4. If a batch is going to be analyzed, which is usually the case, load all samples following the procedure above. After the batch is loaded, replace all samples and standards back in storage.

5. If a dilution is required the following guidelines are followed. If the dilution is > *1/100 (250 uLs of sample) an initial dilution is made into a volumetric flask.* If serial dilutions are required, no less than 1 mL is taken for further dilutions. The final sample aliquot taken for analysis from the volumetric is no less than 250 uLs. If the dilution is < 1/100, the appropriate sample amount is added directly to the 25 mL syringe. In either case, ISS and SSS are added to the 25 mL syringe.

6. Using those parameters listed in Attachment 1, analyze all samples. After analysis, remove the purge vessel from the Tekmar, rinse the purge line and vessel, and place the vessel in the oven to bake at 100°C for at least an hour.

7. Opened sample vials are used only once unless: 1) any necessary dilutions/reruns are done the same day or 2) there are no other vials for that sample.

7.3.2 Soils

1. As some clients still request method 5030 at the present time, soils are still being analyzed as indicated below. As clients convert to Method 5035 completely, this section will be removed.

2. Before weighing any samples, check the balance using the appropriate class weights. Record the actual weights in the Balance Logbook. If a problem is noted, contact the QC department.

3. Allow samples and standards to come to ambient temperature.

4. Weight out 5 grams of the sample into a clean and previously baked purge vessel. Record the weight to 0.1 g. Place the vessel on the Tekmar. Add reagent water to overflowing to a 5 mL syringe. Replace the plunger, invert the syringe, and with tapping, adjust to 5 mLs. Confirm there are no air bubbles. Add 5 uLs of the working ISS/SSS solutions. Transfer the contents to the purge vessel. Using the methods described in Attachment 1, analyze the sample. All soil samples are analyzed with a heated purge (40°C).

5. If a batch of samples is to be analyzed, prepare each as above. After the batch is loaded, replace all samples and standards in storage.

6. For blanks and LCS samples associated with soil analyses, 5 grams of pre-heated sand is weighed into a purge vessel.

7. Any sample that contains targets above the calibration range is diluted to accurately quantitate those compounds. Any sample that, based on historical data has shown to contain high concentrations of compounds is analyzed at an initial dilution. Any sample screening high, is analyzed at an initial dilution. If an initial analysis over-diluted the given sample it is re-analyzed as a low level soil. If the low-level analysis contains compounds above the calibration range, and the same compounds are within range in the dilution, both sets of data may be reported to the client.

8. If a 1/2 or 1/5 dilution is required, 2.5g/1.0g of sample is weighed into the purge vessel.

7.3.3 Medium-Level Soil Extracts

1. If a larger dilution is required, a medium-level soil extract is prepared as follows. Five grams of sample is weighed into a tarred vial. Five (5) mLs of methanol is added to the vial and the vial sealed. A portion of the extract (100 uLs maximum) is taken for analysis. Internal standard and surrogate solutions are added to the 5 mL syringe. Serial dilutions, if needed, are made from the extract and appropriate amounts taken for analysis. A portion is also removed and stored in a 1-1.5 mL Teflon-lined screw-capped vial for storage.

2. If the sample upon which a medium-level prep as been performed also required an MS/MSD, the appropriate amount of MS solution is also added.

3. *All samples prepared in this manner will be analyzed against a medium-level soil* curve. This standards, blanks and LCS samples will contain 100 uLs of methanol. The curve will be at ambient temperature.

NOTE: Some soils are analyzed initially at low levels due to increasing client requests for lower reporting limits. The same samples may then require large dilutions to bring compounds into the calibration range of the instrument. Some compounds, most notably the ketones, have very different responses when heated versus non-heated, despite the sample matrix. Traditionally, the lab heats soils. Therefore, the match between original analyses and dilutions for compounds such as these may not appear to correlate.

Dilution	Sample Weight	Vol. MeOH (1/2.5) Extract
1/2	2.5 grams	---
1/5	1.0 grams	---
1/50	5 grams / 5 mLs	100 uLs
1/250	5 grams / 5mLs	20 uLs
1/500	5 grams / 5 mLs	10 uLs

4. Using those parameters in Attachment 1, analyze all samples in the batch.

5. Sample vials/jars are only used once unless: 1) any dilutions/reruns are analyzed the same day or 2) there is only one jar for analysis.

6. For each new lot number of methanol used, 100 uLs is added to 5 mLs of OFW (organic free water) and analyzed. Absence of target compounds is verified and recorded in the MeOH Lot Check Logbook.

7.3.4 Method 5035

NOTE: ICAL Standards are prepared with 5 mL milli-Q water.

1. Samples for low level VOA soil analysis may be received at the lab in one of two manners: First, as replicate 5 gram core samples in 40 mL vials containing a Sodium Bisulfate preservative solution (refer to USP-5035 for collection/preservation). Alternatively, unpreserved 5 gram core samples may be received in Encore containers. These core samples must be placed in the bisulfate preservative solution within 48 hours of collection. This time requirement is currently under review by appropriate regulatory agencies and may be extended beyond the 48 hours. Until such time, the laboratory will endeavor to "fix" the sample cores in preservative within 48 hours of collection. The laboratory may receive replicate 5 gram soil cores to be used for reanalysis if needed.

2. In addition to low level samples, an additional soil aliquot should be received for use as a screen and possible use as a mid-level extraction/analysis. This additional core must also be fixed in methanol within 48 hours of collection. The amount of methanol added must closely correspond to a soil to solvent ratio of 1:1. Though not specified in the method, STL will pursue a goal of removing the methanol from the soil within 24-48 hours after the initial extraction. A portion of the methanol be removed and placed in an amber 1.5 - 2.0 mL Teflon-lined screw-capped vial for storage. This time limit should standardize the amount of time the methanol comes in contact with the sample.

3. Methanol extracts of soils will be analyzed as stated above at ambient-temperature against a medium-level soil initial calibration. All surrogate and internal standard solutions will be added at time of analysis.

4. Low level soils will be analyzed using the Archon Closed Purge and Trap Auto Sampler System. Surrogate and internal standard solutions will be added at the time of analysis. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and the spike and surrogate tables provided in this SOP. The concentration of the solution and amounts spiked may vary depending on the precision obtained with a given solution/volume combination. However, the final concentrations of such compounds in the samples will follow the same guidelines as previously stated in this SOP for all other samples.

5. As with the internal standard and surrogate, all QC spike solutions must also be added to the closed sample container. This is accomplished by the addition of the spike solutions through the septum with a small gauge (10 uL) syringe just prior to the sample being placed on the instrument for analysis.

6. Some calcareous matrices may react with sodium bisulfate and cause effervescence. The method indicates that such samples need to be recollected without bisulfate preservative and analyzed within 48 hours of collection. Alternatively, some clients and/or regulatory agencies allow optional preservation and holding time criteria. On a per project basis, samples that react with sodium bisulfate may be collected and placed in vials containing water without the preservative. The vials are kept ≤ -12 °C until analysis. Such samples must be thawed prior to analysis. The maximum holding time for this type of collection and preservation is 14-days from the collection date. This alternative approach must be approved by the client/project prior to use.

7.3.5 Drum/Waste Samples

- Non-methanol Miscible
- Methanol Miscible

1. These samples are normally treated as medium level soils or waste dilutions. Waste dilutions normally consist of 1 gram of sample diluted to 10 mLs of methanol. Serial dilutions, if needed, are made from this extract. A portion of the extract is then added to the 5 mL syringe containing surrogate and internal standard. The lab can pre-spike the surrogates and spike compounds if client-specified to do so. As most drum/waste samples result in very high dilutions, it has been the labs experience that surrogates and MSs are most often diluted out and provide no useful information. Unless, specifically assigned to do so, surrogate and matrix spiking will take place at time of analysis.

7.4 Preventive Maintenance

Instrumental maintenance can be categorized as daily and "as required".

7.4.1 Daily Maintenance

The most routinely performed maintenance includes:

- position rinse
- tube baking after sample analysis
- oven bake after high level samples

7.4.2 "As Required"

Most maintenance is done on an "as needed" basis, is operator determined and can be categorized as GC, Tekmar, or MS related.

1. **GC Related**

- change column; condition new column
- clean separator; change separator
- check helium flow rate
- change gas cylinders and moisture trap

2. **MS Related**

- clean source/rods and anything associated with that activity

3. **Tekmar Related**

- rinse positions
- change positions; change parts of positions
- change transfer line; clean transfer line
- replace trap; condition new trap
- refurbish Tekmar
- check purge pressure and flow rate
- analysis of position blanks after high-level samples

- change bulk head fitting

Required maintenance may be performed for a variety of reasons. Certain trouble-flags will indicate what maintenance procedures may be required. A description of the situation, actions taken and follow-up must be documented in the instrument maintenance logbook. An example of the maintenance logbook appears in Attachment 3. In addition, the entry number must be transferred to the appropriate instrument logbook on the day of maintenance, initialed and dated.

7.5 Documentation/Tracking of Sample Analyses

1. The preparation and analysis is recorded in the GC/MS Volatiles Logbook (Attachment 3), and must be completed for each day's analysis.

2. The GC/MS VOA lab employs several forms that serve both a tracking and review function. The Sample Tracking Sheet (BigBoard) is filled out for each job. It contains information the analyst needs as for method, QC requirements, special reporting requirements, etc.., in addition for space to track the analysis of every single sample in the job and the outcome of that analysis.

3. The Tune Form is filled out for every 12 hour tune and contains several kinds of information. The forms main function is to track the analysis of all the samples analyzed during the 12 hours, initial review and data crunching documentation for the samples in the batch, tune and standard information etc.. The Tune Form is not necessarily specific to a single job. The Tune Form is discussed again in the initial review section.

4. In addition, all samples logged into the department appear on a hold-time summary sheet where ALL samples in-house are listed by hold-times and due dates. This summary is utilized by the analysts when making decisions as to methods and analyses that are needed for the day. As samples are analyzed and reviewed, the summary sheet is constantly updated to reflect those samples completely analyzed, those requiring dilutions and re-analyses (essentially a posted summary of the Sample Tracking Sheets). At the beginning of each day, the completed analyses are removed from the summary sheet by updating the analyzed samples into LabNet. The Sample Tracking Sheet and Tune Form can be found in Attachment 3.

7.5.1 Archival of Data

There are three full back-ups performed per week.

- Every Thursday a full back up of VOA data is performed.
- Every Friday a full back up of SVOA data is performed.

- Every Tuesday a system back up (minus the NBS Library) is performed. There are two tapes provided for this back up, and are rotated each week. Most current tapes are kept off site. Older tapes are in locked storage.

The system maintains a database, or logs, of each back-up session. Successful completion of each back-up can be verified each morning by accessing the job report logs in ARCServe. This is done each morning. Any missed jobs can be rescheduled and completed in the morning of the following day. As noted above, this database is re-archived after every normal back-up and can be retrieved at any time necessary.

7.5.2 Removal of Data

1. Although there is a substantial amount of space available to both BNA's and VOAs during busy periods the system can fill rather quickly. As an estimate, with a total of twelve (12) instruments, a maximum of about 2-3 months (per instrument) can be kept on the system at one time. There is not necessarily a set definite schedule of removing data from the system. As per laboratory SOP's, once the data package has been removed and all data associated from that batch has been reduced, reviewed, packaged and sent to report generation, the tune form is placed in designated location. Either by necessity or at the supervisors discretion, these are compiled and the data then actually removed from the system.

2. The tune forms are then filed in the office area. Once a year, these forms are compiled and boxed and stored in a general data storage area.

8.0 QUALITY CONTROL

8.1 QC Summary

The department will review the quality controls as follows:

8.1.1 Method Blank (MB) / Laboratory Control Standard (LCS)

At least one MB and LCS will be included in each laboratory batch. Regardless of the matrix being processed, the LCS and MBs will be in an aqueous media.

The MBs will be examined to determine if contamination is being introduced in the laboratory. The LCS will be examined to determine accuracy and precision.

8.1.2 Accuracy

Accuracy will be measured by the percent recovery (%R) of the LCS. Method 8260B list or suggest accuracy limits for an initial demonstration of precision and accuracy. There are no further guidelines for spike recoveries. The current limits for the suggested spike compounds listed in the methods are listed in Attachment 2 of this SOP. Internally, QA/QC will monitor %R, and will plot control charts to monitor method accuracy and generate control limits when deemed necessary. The number of compounds being used for bench level control and the accuracy limits assigned to those compounds may vary with client, QAP, project etc.. This information is transmitted to the bench via the COC, kick-off meetings, tech profiles etc.., and indicated on one of the forms used at the bench. The accuracy limits are also posted on controlled boards in both the analytical and data areas (includes both method/sop limits and in-house generated limits. In-house generated limits are subject to change, but are included in Table 1).

8.1.3 Precision

Precision will be measured by the reproducibility of the LCS and will be calculated as Relative Percent Difference (RPD). The Methods list guidelines for the initial demo as noted above, but give no further guidelines for spike data. Current limits are listed in Table 1, however, RPD's are not used to assess bench level CA prior to sample analysis. Internally, QA/QC will monitor precision and calculate limits and log in LCS and LCD's at the appropriate time, at which both will be analyzed. Otherwise, only one LCS will be completed.

8.1.4 Surrogates

Surrogate Compounds will be added to every sample to measure performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable. Limits are listed in Table 1. In-house surrogate limits are also generated as per method. As with LCS samples, the limits used to assess accuracy vary with client, QAP, project etc.., and the information transmitted to the bench in the same manner.

8.1.5 QC Charting

Precision and accuracy are monitored using LCS data. In-house criteria have been generated and are in use at present. Additional data may be added at QA/QC discretion during the year. During that period of time, additional LCSs will be logged into the system until adequate data are generated. Spike levels are 50 ppb/10 ppb for soils/waters. Only those compounds listed above are spiked and should be representative of the whole. The more non-routine compounds are not part of the spiking solutions. Other limitations (availability of second source) may also prevent adding these to spiking solutions. See comments in Section 6 for application of in-house control limits.

8.2 Corrective Actions

Listed below are the steps to be taken when an out-of-control situation occurs. The analyst must address the following issues as described below in the individual sections.

- demonstrate that all of the problems creating the out-of-control situation were addressed;
- document the problem and the action that was taken to correct the problem;
- document that an in-control situation has been achieved; and
- receive approval (signature) of the supervisor, section manager, QC personnel or other qualified personnel prior to release of data associated with the problem.

Bound corrective action (CA) logs are located in each run-log. In addition, a separate CA form, specific to a unique job, is attached to the COC and sample tracking form when the samples are initially entered into the sample tracking documentation used by the department. The log-book and sample # are used to note all out-of-control events, the actions taken to try and correct the problem, the return to control and qualification of data is needed.

Discussed below are the suggested and required courses of action when an out-of-control situation has occurred.

8.2.1 Surrogates

All surrogate recoveries are calculated. If ANY surrogates are outside limits in the MB, it must be re-analyzed. Analyses CAN NOT proceed until an in-control situation is demonstrated. Re-analyze the blank. If surrogates are still out, the instrument may need to be re-tuned (BFB) and/or another calibration standard analyzed. If the problem persists, further maintenance action may be required (i.e., trap replacement, clean *separator).*

Before pursuing other measures, check to be sure that:

- calculations are correct
- concentrations of the surrogates in the spiking solution are correct
- the correct amount of ISS/SSS *solution was added*
- ISS/SSS areas are reasonable

If any surrogates in a sample are outside limits, check the above first. Any sample that has a surrogate out must be re-analyzed. The re-analysis can take the form of a dilution, if there is reasonable expectation that a high concentration of a target compound is causing a matrix effect. If the surrogate(s) is/are still outside limits, a matrix effect is demonstrated and both reports are submitted. Depending on the client, the best result may be reported and the other result narrated. If all surrogates are in-control on the re-analysis, only the second analysis is reported.

Every effort is made to complete the re-analysis within hold-time. If this is impossible (i.e. , capacity hold-times preclude re-analyses hold-time)m both reports may be submitted. This is documented in the narrative.

If the sample with the out-of-control surrogates is the same sample on which the MS and MSD has been performed, and the pattern is duplicated, then re-analysis is NOT required. Documentation of the similarities is required.

Surrogate corrective action is documented on the Individual CA Sample # Report for samples, and in the CAR logbooks for blanks and LCS samples.

8.2.2 BFB Criteria

If BFB criteria can not be met, determine if the source of the problem is instrumental or tune related. Inspect overall sensitivity, possible excessive background, the proportionality of the masses, relative abundances of the target masses. If it seems tune-related, adjust the tune parameters in Manual Tune slightly, until acceptance is achieved. If the problem seems instrumental, perform suggested trouble-shooting to locate and correct the problem (Suggestions can be found in most of the manuals). NO analysis can proceed until criteria are met. Corrective action for BFB analysis is documented in the CA logbook associated with the instrument in question. 'Return to control' must be documented.

8.2.3 Initial Calibration

If initial calibration can not be met, determine if the problem is analytical or instrumental. Some suggested questions to ask would be:

- were the standards prepared correctly?
- was the proper amount analyzed?
- check the chromatogram - did something happen on one or two analyses; i.e., a leak
- check the response factors - is one concentration level very high or low? re-analyze
- how old are the standards?

All calibration criteria must be met (Section 6.4). If the ICAL does not meet specified criteria, at minimum, the appropriate levels must be re-analyzed. If necessary, new standards should be prepared and the levels re-analyzed. During analysis of an initial calibration, documentation of the re-analyses of specific levels is not required. See previous section outlining CA for minimum COD values as well.

8.2.4 Continuing Calibration

If continuing calibration can not be met, determine if the problem is analytical or instrumental. Some suggestions:

- check the chromatography
- is overall sensitivity low?
- excessive background?
- how old is the standard?
- need a new 5-point?
- has the tune shifted?

Compare the relative abundances of 69, 131 and 219 from that days manual tune to those on the day the initial calibration was analyzed. Slight adjustments to the tune may bring the standard in. Certain compounds will help indicate what the problem is.

All calibration criteria must be met (Section 6.5). If the CCAL does not meet specified criteria, at minimum the standard should be re-analyzed. A new standard may be prepared and then re-analyzed. If necessary, a new ICAL must be run. All action taken for CCAL's must be recorded in the CAR logbook for that instrument. 'Return to control' must be documented.

8.2.5 Method Blank (MB)

If the MB is/appears to be contaminated, re-analyze it on a different position. If contamination is still present, the problem may be in one of the common elements, such as the trap, transfer line, port valve or column. Baking the trap/column and running position blanks may be necessary. If contamination has occurred beyond that, and maintenance is required (i.e., replace trap) it is documented in the Maintenance Logbook. Corrective action and return to control for MBs is recorded in the CAR logbook for the appropriate instrument. Under extenuating circumstances, if analysis continues, qualification must be made as to the positive hits above the EQL for the compounds in question. Any associated samples analyzed in the tune must be noted. Any samples containing positive hits must be noted. IF, the samples containing positive hits can not be re-analyzed (i.e., past hold-time), the positive hits are flagged with "B" and the situation and data noted and qualified in a case narrative.

8.2.6 Laboratory Control Sample (LCS)

As specified in Section 8.1.2, the number of compounds and the limits used to assess accuracy vary with client, QAP, project etc.. Both method/SOP and in-house generated limits are listed in the Appendices. The in-house limits are subject to change. The need and course of corrective action varies with the number of compounds being used for bench control and positive detected of compounds outside limits. The following guidelines are used :

(1) AFCEE : All compounds are used for bench control.

- If any compound exhibits low recoveries, the LCS is re-analyzed. If the compound is still low a new spike may be prepared and the LCS re-analyzed. Analysis should not continue until the situation is taken care of. All corrective action is documented at the time and return to control demonstrated for low compounds. IF, in <u>extenuating circumstances</u>, analysis is continued, the low compounds must be noted in the CAR book, associated samples must be qualified in the qualification section, absence of CA documented etc.. Data must be qualified for those compounds in the narrative.

- If any compound exhibits high recoveries, the LCS may be re-analyzed, and/or a new solution prepared, and/or a new standard prepared and calibrations repeated.

All corrective action is recorded at the time in the CAR logbook and return to control documented if applicable. AFCEE allows for high recoveries on a one time basis if the said compounds are not detected in the associated samples. Any high compounds must be noted in the CAR logbook, the associated samples listed in the qualification section, and the presence or absence of these compounds in the associated samples. If positive detects are noted, and the samples are unable to be re-analyzed, the situation must be documented and noted in the case narrative. Following the first occurrence of high recoveries, the bench will take appropriate note and follow-up with appropriate CA within a reasonable amount of time.

(2) QAP's etc.., specifying five compounds.

- ALL five compounds must be within limits for analysis to proceed. The LCS samples may be re-analyzed. New spike solutions may be prepared. Or new standards or CCAL's may be analyzed. All corrective action and return to control must be documented at the time in the CAR logbook of the appropriate instrument.

- The actual limits used for the five compounds may be QAP specific (usually those listed in the table in the appendix) or in-house generated by matrix and method. In either case, the above CA and required documentation apply.

- For all other compounds in the full-list spike, all recoveries are assessed, although no immediate corrective action may be required. If the recoveries are low, in general another LCS may be re-analyzed. The spike solution and standard may be verified for correct concentrations. However, no corrective action is absolutely required by the bench unless an error is discovered. The recoveries may or may not be documented in the narrative, however, they are noted on the review form. The recoveries of the "un-controlled" compounds may be used for data interpretation.

- Although not strictly required to take immediate corrective action, the purpose of the full-spike is two-fold in that the bench should use it as an indicator of the status of the calibration standards, instrument conditions etc.., as well as a tool for data interpretation. Therefore, in keeping with good lab practice, the situation should be noted and assessed and any corrective action deemed necessary should be taken within a reasonable amount of time (Example: High recoveries on gases => new calibration standard may be needed).

8.2.7 Matrix Spikes (MS)

As specified in Section 8.1.2, the number of compounds and the limits used to assess accuracy vary with client, QAP, project, etc.. Both method/SOP and in-house generated limits are listed in the Appendices. The in-house limits are subject to change. The need and course of corrective action varies with the number of compounds being used for bench control and recoveries of same compounds in the associated LCS samples. The following guidelines are used :

(1) AFCEE : All compounds are used for bench level control.

- If the MS exhibits recoveries outside limits, AFCEE requires it to be re-analyzed as the MSD. No further action is required. Documentation is required however, on the individual CAR form and association made to the LCS for those compounds and in the case narrative. See above specifications for associated LCS samples.

(2) QAP's etc.., specifying five compounds.

- ALL five compounds are assessed. If recoveries are outside limits, the LCS is reviewed for those compounds. If the recoveries are within limits in the associated LCS samples, no further action is required. See above section concerning LCS CA for further information and action required for recoveries outside limits in LCS samples.

- The actual limits used for the five compounds may be QAP specific (usually those listed in the table in the appendix) or in-house generated by matrix and method. In either case, the above CA and required documentation apply.

- For all other compounds in the full-list spike, all recoveries are assessed, although no immediate corrective action may be required. The affected compounds may be compared to the same compounds in the associated LCS samples. See the above section for further information and action required for these compounds in the LCS samples. The recoveries may or may not be documented in the narrative, however, they are noted on the review form. The recoveries of the "un-controlled" compounds may be used for data interpretation.

8.2.8 Internal Standard Policy

Method 8260 does not require re-analyses of samples for low internal standard areas. However, it is STL's policy to monitor areas and retention times, therefore, the following guidelines apply.

Situations requiring re-analyses :

- If ALL areas are outside limits the sample will be re-analyzed.
- Any sample that has a positive hit associated with any internal standard outside limits will be re-analyzed.
- If ANY surrogates are outside limits the sample will be re-analyzed.

Situations NOT requiring re-analyses :

- If all surrogates are within limits and there are no positive hits associated with those internal that are outside limits, the sample does not have to be re-analyzed. Situation should be addressed in the case narrative and noted on the sample CAR form.
- If all surrogates are within limits, but there is an obvious matrix effect occurring, even if positive hits are noted, the sample does not need to be re-analyzed. This decision will be approved by the supervisor or section manager. The situation must also be addressed on the sample CAR form and narrative.
- If there is historical evidence that shows a repeated pattern for a certain client and site, and this can be documented by reviewing past projects, the samples do not have to be re-analyzed. This decision will be approved by the supervisor or section manager.
- Internal standard areas for samples are documented on the Individual CAR form. Internal standard areas for CCAL to CCAL are documented in the appropriate CAR logbook.

Any sample showing retention times outside windows will be re-analyzed. This is documented in the appropriate manner as in the preceding paragraph.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Computer Data Production/Reduction

The Target 3.5 software produces a Total Ion Chromatogram (TIC), header, quant report and background subtracted spectra. For those clients requiring it, a 5 tentatively identified compound (TIC) search is also performed. The data system will produce an integration listing and tentative identification of each hit found at the selected percentage of the largest peak present.

9.1.1 Quantitation of Target Compounds

Quantitation of the target compounds is performed by the data system can be accomplished as follows:

WATERs: concentration (mg/L) = $\frac{[A_x \times I_s]}{[A_{is} \times RF]} \times DF$

Where:
A_x = area of characteristic ion for target
I_s = concentration of internal standard (ng)
A_{is} = area of characteristic ion for int. std.
RF = response factor for target
DF = dilution factor (if any)

SOILs: concentration (mg/kg) = $\frac{[A_x \times I_s]}{[A_{is} \times RF \times D]} \times DF$

Where:
All variables are equal and
D = (100 - % moisture in sample/100) or 1 for wet weight. (As in the case of drum samples)

The target methods all contain calculations for waters and soils that allow automatic processing and calculations of concentrations to be completed. The user may enter some variables (Dilution Factor) and others are imported from LabNet. Sample prep info for VOA's is entered directly into LabNet. The sample volume is considered to be "constant" for calculation purposes. Less sample volume (in the case of waters) and soil weight (in the case of soils) are taken into account in the dilution factor entered by the user. For medium-level soils and waste/drum type samples medium level calculations are needed and actual weights are brought into LabNet.

NOTE: As noted previously, weights are recorded to 0.1 gram. It is SOP to weigh out 5.0 grams (or as appropriate for the dilution), however, to keep data entry and calculations simple. The same holds true for all water volumes.

9.1.2 Accuracy: $\%R = \frac{(A_T - A_0)}{A_F} \times 100$

Where:
A_T = Total amount recovered in fortified sample
A_0 = Amount recovered in unfortified sample
A_F = Amount added to sample

9.1.3 Precision: $\% D = \frac{|B_1 - B_2|}{B_1} \times 100$

$$RPD = \frac{|B_1 - B_2|}{(B_1 + B_2)/2} \times 100$$

Where:
B_1 = %Recovery MS (or LCS)
B_2 = %Recovered MSD (or LCS)

9.1.4 Modifications for 8260B quantitation

1. **Initial Calibration Criteria**

Methods 8000B/8260B require the use of linear or higher order calibration curves for those compounds exceeding 15%.

The following equations apply :

Linear Regression :

$$y=a_0+(1/a_1)x$$

Quadratic Curve:

$$y = a_o + (a1 * x) + (a2 * x^2)$$

Power Curve :

$$y=e^{a0} * x^{(1/a1)}$$

which is converted to :

$$\ln(y) = (1/a_1) * \ln(x) + a_0$$

$x = Area_{UKN}/Area_{ISTD}$
$y = Amount_{UNK}/Amount_{ISTD}$

Once the $Amount_{UNK}$ is solved, the value is adjusted for total solids, dilution factors etc.., to calculate a final concentration.

The quantitation of compounds using either a linear regression, quadratic curve or a power curve as performed automatically by the Target software has been confirmed to be accurate.

Method 8000B/8260B specifies a minimum COD.

The corrective action regarding an initial calibration for method 8260B as it relates to the 0.990 correlation coefficient acceptance criteria is outlined. When a compound has a correlation coefficient less than 0.990, the occurrence is documented by the analyst in the GC/MS VOA CAR section of the instrument's logbook. Any corrective action or data qualification is also documented in the CAR section of the logbook. All corrective actions taken may include those listed below.

Samples may be analyzed against an initial calibration that have compounds with a correlation coefficient less than 0.990 and the corrective actions taken may also include some but not all of the following:

- The data for these samples may be reported without qualification if the compounds with a correlation coefficient less than 0.990 are not detected in the sample, therefore no further corrective action is required.
- If a compound is detected in the sample that has a correlation coefficient less than 0.990, the samples may be reanalyzed against an initial calibration with an acceptable correlation coefficient and only the reanalysis will be reported on the sample. If this reanalysis occurs beyond analysis hold times then both analyses on the sample will be reported.
- If a compound is detected in the sample that has a correlation coefficient less than 0.990, the decision to reanalyze or to reported the data without further corrective action is made on a case by case basis with the approval of the supervisor, the section manager, the project manager and the client. The sample results may require qualification for this compound on the report and will be addressed in the case narrative.

2. **Continuing Calibration Check**

Prior to sample analysis a 10 ppb/ 50 ppb calibration check is completed. All minimum RF's must meet same limits. All CCC's must be less than 20% Drift as calculated below; the analysts may verify %DIFF and only calculate those that are close. (Error may only be made in favor of tighter control).

$$\%Drift = \frac{(Ci - Cc)}{Ci} \times 100$$

Where:
Ci = standard conc. (10/50)
Cc = measured conc. in cal check

9.1.5 Quantitation of TIC's (Tentatively Identified Compounds)

Quantitation of TIC's is performed by the Target processing software. The formulas above for waters and soils can be used with the following modifications. A_x and A_{is} should be taken from the total ion integration listing accompanying the TIC report produced by the data system. The nearest non-interfered with internal standard should be used. The RF is assumed to be one (1). The concentration is therefore an estimate and is flagged as such with a "J". Any TIC also found in the MB is flagged with a "JB". Any TIC identified with a CAS number is also flagged with an "N", indicating that the ID was based on the mass spectra. The operator should visually confirm that the integration is correct. If not, the peak in question must be manually integrated. The target system automatically calculates the actual concentration of the TIC's, including dilutions and total solids, once that information is retrieved from LabNet.

9.2 Operator Data Reduction/Review

The operator does on-screen review of all data and 1) makes judgments concerning the "realness" of those target compounds found and 2) makes judgments concerning the identification of the tentatively identified compounds and 3) modifies the output to produce a data package reflective of those decisions.

9.2.1 Initial Review

The GC/MS VOA area uses two kinds of corrective action documentation. The first consists of the CAR section of logbooks, that are specific to each instrument. These logbooks contain sections to report out-of-control situations, CA, return-to-control and qualification sections for documenting problems related to general QC: tune, ICAL, CCAL, internal standard areas from CCAL to CCAL, and LCS samples. The second are the Individual CAR's that refer to a single job. These are used to record events, CA and final actions for surrogates, internal standard areas, carry-over situations, analyses past tune time, MS/MSD data etc.., for each sample in the batch. These forms are attached to the COC, along with the samples tracking sheets. Both may be used during initial review of the data. Examples of both can be found in Attachment 3. See Section 8.2 of this SOP for details on CA.

All data is initially reviewed on-screen. The review is both a QC review and a general review as described below.

- The MB contains no interferences or target compounds at the EQL.
- ALL surrogates are in control in the blank. Surrogate limits are listed in Table 1;
- ALL surrogates in samples are in control;

- LCS recoveries meet the limits listed in Table 1. See Section 8.2 concerning compounds and limits for LCS samples. In-house limits have been generated and are in use.
- Internal standard areas and retention times are checked and meet guidelines. Limits are listed in Attachment 2. Additional guidelines can be found in Section 8.2.
- The sample does not require any further dilutions or analysis at a more concentrated level. Dilutions are made to keep the target in the upper half of the calibration range. The MS and MSD are never diluted to get spiked or non-spiked compounds within range, as this would reduce the matrix affect assessment.
- Visually confirm complete integration for any large and/or saturated target compounds.
- The sample does not require re-analysis for any other reason (i.e., leak, analysis past tune time, ISTD areas low, etc..).

9.2.2 Identification of Targets

The following guidelines are used in the positive identification of target compounds.

1. "elution of component at the same relative retention time as the standard component."

The elution times should compare within +- 30 s. The standard must be run on the same 12 hour period as the sample. If co-eluting analytes interfere with the comparisons of retention times, other ions characteristic to that compound can be used to confirm relative retention times.

2. "correspondence of the sample component and standard component mass spectrum." Comparisons of sample spectra to standard spectra must be made using standard spectra obtained from the GC/MS system.

All ions present in the standard spectrum at a 10% relative intensity (most abundant ion being 100%) should be present in the sample.

The relative intensities of the above ions should agree within ±20%, between the standard and sample. If an ion is 50% intensity in the standard the corresponding ion must be between 30 and 70% in the sample.

Ions >10% in the sample but not present in the standard should be considered and accounted for. (A user program exists to aid in this).

3. **Operator judgment**. If a compound can not be verified by the above, but in the operators technical judgment the ID is correct, it is reported as such.

4. Once all positive identification is made, the file is modified to reflect these decisions. At this time TIC's may also be reviewed and name. In each case where the file has been edited or manual integrations have taken place the operator must identify, initial and date the changes on the hardcopy. The following guidelines apply :

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Manual integrations are automatically flagged with an "M" on the raw data.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.

9.2.3 Manual Integration Policy

In each case where the file has been edited or manual integrations have been performed the operator must identify, initial, and date the changes on the hardcopy report. The following guidelines apply:

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Manual integrations are automatically flagged with an 'M' on the raw data.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.
- Manual integrations shall follow the STL Corporate SOP for manual integrations (#S-Q-004).

Manual integrations are most often performed for the following reasons:

- Assignment of correct peak that was mis-identified by the data system.
- Incomplete auto-integration due to high level of target compound detected.
- Incomplete auto-integration due to background interference.
- Incorrect auto-integration due to co-elution or near co-elution of compounds.
- Missed peaks.

The analysts review all integrations. Spectra and Extracted Ion Chromatography Profiles (EICP) are printed after any integration takes place for target compounds and are routinely included in the data packages. If EICP's for internal standards, surrogates and calibration standards are required to satisfy client deliverables, they can be provided, but will not be routinely added. Manual integrations may be documented in the case narrative if so required, however, references to this SOP will be used for explanations, and any further documentation beyond initials and dates will not be done.

9.2.4 Identification of TIC's

In general, up to as many as 5 non-target compounds are tentatively identified by the data system and operator. Compounds with responses >10% of the nearest ISTD are identified. The data system provides the operator with a SUB ADC C sample spectrum, spectra of the first three matches and a listing of two other possibilities. Molecular formulas, molecular weights and CAS #'s are included. The following guidelines are used:

Relative intensities of major ions in the reference spectrum should be present in the sample (ions >10%).

- Relative ions should agree within $\pm$20%;
- Molecular ions in the reference should be in the samples;
- Review the possibility of background and/or co-eluting compounds for those ions present in the sample but not in the standard;
- If ions are present in the sample but not in the standard, review the possibility of the presence of background or co-eluting compounds;
- If ions are present in the standard but not in the sample, review the possibility that the ions were subtracted out because they are also common to the background or co-eluting compounds;
- In the event no valid interpretation can be made, the compound is called "unknown".
- Interpretation can be often narrowed down to a class of compounds, molecular formula or weight.

9.3 Final Review

1. Once 1) the analysis is determined to be acceptable and 2) the initial review and data reduction has occurred (verifiable on the Tune Form) and 3) the analyst has entered sample prep info into LabNet, the following steps occur. The sample prep information, client ID information and some data applicable to fields in the forms is retrieved from LabNet. At this point, the analyst then usually prints hard-copies of all the necessary raw data. The analysts review the hard-copies and initial and date them, documenting that review.

2. All required forms are then generated using the Target software. Most, if not all of these steps, are documented on the tune form. The package is then assembled and ready for the first review.

3. Upon the first 100% review, the review form is initialed and dated as reviewed. The initial review is normally done by the analyst preparing the data package. The package, with its review sheet, comments and any CAR forms is submitted to the supervisor or section manager for a second review and validation. Once the data passes review in the department, it is submitted to report generation/QA/QC for appropriate follow-up action.

4. The complete analysis scheme can be summarized below (Section 7.1.1 & 7.1.2) and in Attachment 5. The entire sample tracking system can be summarized in Attachment 5. The Tune Form is used to verify many of these steps of review and data reduction.

10.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- Waste from this procedure will enter the 'Flammable Vials' wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Table 1. Estimated Quantitation Limits for Volatile Analytes; Laboratory Statistical Control Limits
Table 2. Characteristic Mass for Purgeable Organics Compounds

Figure 1. Example: Total Ion Chromatogram for 25 mL Purge Water
Figure 2. Example: Total Ion Chromatogram for 5 mL Purge Soil

Attachment 1. Example: Method Listings; Tekmar Conditions; Flow Settings
Attachment 2. Example: Target and Internal Standards; ICAL/CCAL; Surrogate Recovery Limits; LCS / MS Recovery Limits; Internal Standard Guidelines
Attachment 3. Example: GC/MS Volatiles/CAR Logbook; Tune Form; Sample Tracking Sheet; Maintenance Logbook
Attachment 4. Calibration Evaluation and Acceptance Criteria
Attachment 5. Example: Analysis and Sample Tracking Flowcharts
Attachment 6. Example: Data Review Form

Historical File:

Revision 00: 06/22/92	Revision 07: 04/10/97
Revision 01: 04/26/93	Revision 08: 06/13/97
Revision 02: 05/26/93	Revision 09: 02/11/98
Revision 03: 01/24/94	Revision 10: 01/29/99
Revision 04: 07/15/94	Revision 11: 03/08/99
Revision 05: 11/20/95	Revision 12: 09/28/00
Revision 06: 04/09/96	Revision 13: 10/21/02

Reasons for Change, Revision 13:

- Annual review.
- Updated the Health & Safety (3.0) and Waste Disposal (10.0) sections.

U:\QC\SOP\UMV\8260.DOC

Table 1.

Example: Estimated Quantitation Limits for Volatile Analytes[a]
Laboratory Statistical Control Limits

[a]Estimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following example information for further guidance on matrix-dependent EQLs.

[b]EQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

STL Chicago

Method Limit Report

Project:

Updated: 3/4/02

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Method: Volatile Organics (5mL Purge) (8260.5)										
1,1,1,2-Tetrachloroethane	8260B	Water-5mL	ug/L	0.96	5	78	119	20		
1,1,1-Trichloroethane	8260B	Water-5mL	ug/L	1.2	5	69	130	20		
1,1,2,2-Tetrachloroethane	8260B	Water-5mL	ug/L	1.7	5	72	121	20		
1,1,2-Trichloroethane	8260B	Water-5mL	ug/L	1.5	5	66	135	20		
1,1-Dichloroethane	8260B	Water-5mL	ug/L	1.3	5	56	124	20		
1,1-Dichloroethene	8260B	Water-5mL	ug/L	1.7	5	48	132	20		
1,1-Dichloropropene	8260B	Water-5mL	ug/L	1.1	5	57	150	20		
1,2,3-Trichlorobenzene	8260B	Water-5mL	ug/L	4.3	5	70	126	20		
1,2,3-Trichloropropane	8260B	Water-5mL	ug/L	1.2	5	68	124	20		
1,2,4-Trichlorobenzene	8260B	Water-5mL	ug/L	3.3	5	57	132	20		
1,2,4-Trimethylbenzene	8260B	Water-5mL	ug/L	1.1	5	69	128	20		
1,2-Dibromo-3-chloropropane	8260B	Water-5mL	ug/L	1.8	5	51	119	20		
1,2-Dibromoethane (EDB)	8260B	Water-5mL	ug/L	1.3	5	69	124	20		
1,2-Dichlorobenzene	8260B	Water-5mL	ug/L	1.5	5	80	117	20		
1,2-Dichloroethane	8260B	Water-5mL	ug/L	1.4	5	68	125	20		
1,2-Dichloroethene (total)	8260B	Water-5mL	ug/L	2.4	5	58	129	20		
1,2-Dichloropropane	8260B	Water-5mL	ug/L	1.1	5	67	124	20		
1,3,5-Trichlorobenzene	8260B	Water-5mL	ug/L	2.4	5	67	118	20		
1,3,5-Trimethylbenzene	8260B	Water-5mL	ug/L	1.1	5	68	126	20		
1,3-Butadiene	8260B	Water-5mL	ug/L		5					
1,3-Dichlorobenzene	8260B	Water-5mL	ug/L	1.5	5	76	118	20		
1,3-Dichloropropane	8260B	Water-5mL	ug/L	1.1	5	72	121	20		
1,4-Dichlorobenzene	8260B	Water-5mL	ug/L	1.6	5	78	119	20		
1-Chlorohexane	8260B	Water-5mL	ug/L	0.9	5	60	143	20		
2,2-Dichloropropane	8260B	Water-5mL	ug/L	4.5	5	56	139	20		
2-Butanone (MEK)	8260B	Water-5mL	ug/L	1.9	5	55	140	20		
2-Chloro-1,3-butadiene (chloroprene)	8260B	Water-5mL	ug/L	0.29	5					
2-Chloroethylvinylether	8260B	Water-5mL	ug/L	2.3	5	10	150	20		
2-Chlorotoluene	8260B	Water-5mL	ug/L	1.1	5	75	123	20		
2-Hexanone	8260B	Water-5mL	ug/L	1.4	5	50	136	20		
2-Methylnaphthalene	8260B	Water-5mL	ug/L		5					
2-Nitropropane	8260B	Water-5mL	ug/L		400					
3-Chloropropene (Allyl Chloride)	8260B	Water-5mL	ug/L	1.4	10					
4-Chlorotoluene	8260B	Water-5mL	ug/L	1.2	5	69	122	20		
4-Methyl-2-pentanone (MIBK)	8260B	Water-5mL	ug/L	1.3	5	50	138	20		
Acetone	8260B	Water-5mL	ug/L	4.8	5	33	141	20		
Acetonitrile	8260B	Water-5mL	ug/L	20.2	40					

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Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Acrolein	8260B	Water-5mL	ug/L	74.5	200					
Acrylonitrile	8260B	Water-5mL	ug/L	16.3	40					
Benzene	8260B	Water-5mL	ug/L	0.96	5	68	130	20		
bis(chloromethyl)ether	8260B	Water-5mL	ug/L		2000					
Bromobenzene	8260B	Water-5mL	ug/L	1.2	5	73	127	20		
Bromochloromethane	8260B	Water-5mL	ug/L	1.5	5	64	135	20		
Bromodichloromethane	8260B	Water-5mL	ug/L	1.3	5	69	131	20		
Bromoform	8260B	Water-5mL	ug/L	1.1	5	70	128	20		
Bromomethane	8260B	Water-5mL	ug/L	0.86	5	68	144	20		
Carbon disulfide	8260B	Water-5mL	ug/L	1.6	5	50	150	20		
Carbon tetrachloride	*8260B*	*Water-5mL*	*ug/L*	*0.89*	*5*	*66*	*121*	*20*		
Chlorobenzene	8260B	Water-5mL	ug/L	1	5	77	121	20		
Chloroethane	8260B	Water-5mL	ug/L	1.3	5	48	177	20		
Chloroform	8260B	Water-5mL	ug/L	1.6	5	66	127	20		
Chloromethane	8260B	Water-5mL	ug/L	2.7	5	62	131	20		
cis-1,2-Dichloroethene	8260B	Water-5mL	ug/L	1.3	5	62	134	20		
cis-1,3-Dichloropropene	8260B	Water-5mL	ug/L	1.2	5	73	127	20		
Cyclohexanone	8260B	Water-5mL	ug/L		400					
Dibromochloromethane	8260B	Water-5mL	ug/L	1.1	5	74	119	20		
Dibromomethane	8260B	Water-5mL	ug/L	1.3	5	65	124	20		
Dichlorodifluoromethane	8260B	Water-5mL	ug/L	1.2	5	41	127	20		
Ethyl acetate	8260B	Water-5mL	ug/L	10.2	50					
Ethyl ether	8260B	Water-5mL	ug/L	0.38	5	35	121	20		
Ethylbenzene	8260B	Water-5mL	ug/L	0.93	5	78	121	20		
Ethylmethacrylate	8260B	Water-5mL	ug/L	0.99	10					
Heptane	8260B	Water-5mL	ug/L	0.59	5	17	124	20		
Hexachlorobutadiene	8260B	Water-5mL	ug/L	3.5	5	58	133	20		
Hexane	8260B	Water-5mL	ug/L	0.66	5	36	76	20		
Iodomethane	8260B	Water-5mL	ug/L	1.1	10	50	150	20		
Isobutyl alcohol	8260B	Water-5mL	ug/L	122	400					
Isopropyl alcohol	8260B	Water-5mL	ug/L							
Isopropyl ether	8260B	Water-5mL	ug/L	0.32	5					
Isopropylbenzene	8260B	Water-5mL	ug/L	0.87	5	72	118	20		
m&p-Xylenes	8260B	Water-5mL	ug/L	1.8	10	73	128	20		
Methacrylonitrile	8260B	Water-5mL	ug/L	1.8	10					
Methylene chloride	8260B	Water-5mL	ug/L	1.4	5	45	130	20		
Methylmethacrylate	8260B	Water-5mL	ug/L	1.5	10					
Methyl-tert-butyl-ether (MTBE)	8260B	Water-5mL	ug/L	2	5	49	139	20		

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Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Naphthalene	8260B	Water-5mL	ug/L	3.6	5	60	119	20		
n-Butyl alcohol (1-Butanol)	8260B	Water-5mL	ug/L	400	400					
n-Butylbenzene	8260B	Water-5mL	ug/L	1.5	5	71	128	20		
n-Propylbenzene	8260B	Water-5mL	ug/L	0.96	5	70	124	20		
o-Xylene	8260B	Water-5mL	ug/L	0.98	5	75	123	20		
Pentachloroethane	8260B	Water-5mL	ug/L	1.1	10					
p-Isopropyltoluene	8260B	Water-5mL	ug/L	1	5	75	126	20		
Propionitrile	8260B	Water-5mL	ug/L	14.5	40					
sec-Butylbenzene	8260B	Water-5mL	ug/L	0.96	5	74	124	20		
Styrene	8260B	Water-5mL	ug/L	1.1	5	80	126	20		
tert-Butyl alcohol	8260B	Water-5mL	ug/L							
tert-Butylbenzene	8260B	Water-5mL	ug/L	0.98	5	73	123	20		
Tetrachloroethene	8260B	Water-5mL	ug/L	2.6	5	74	123	20		
Tetrahydrofuran	8260B	Water-5mL	ug/L	1.8	5	22	147	20		
Toluene	8260B	Water-5mL	ug/L	1.1	5	72	122	20		
trans-1,2-Dichloroethene	8260B	Water-5mL	ug/L	1.1	5	63	125	20		
trans-1,3-Dichloropropene	8260B	Water-5mL	ug/L	1.4	5	62	127	20		
trans-1,4-Dichloro-2-butene	8260B	Water-5mL	ug/L	1.1	10					
Trichloroethene	8260B	Water-5mL	ug/L	1.6	5	71	128	20		
Trichlorofluoromethane	8260B	Water-5mL	ug/L	3.2	5	68	133	20		
Trichlorotrifluoroethane	8260B	Water-5mL	ug/L	1	5	50	150	20		
Vinyl acetate	8260B	Water-5mL	ug/L	1.4	5	58	157	20		
Vinyl chloride	8260B	Water-5mL	ug/L	1.2	5	50	141	20		
Xylenes (total)	8260B	Water-5mL	ug/L	1.8	5	76	129	20		
Surrogate										
1,2-Dichloroethane-d4 (surr)	8260B	Water-5mL	ug/L						66	132
4-Bromofluorobenzene (surr)	8260B	Water-5mL	ug/L						79	122
Dibromofluoromethane (surr)	8260B	Water-5mL	ug/L						66	132
Toluene-d8 (surr)	8260B	Water-5mL	ug/L						78	128
Method: Volatile Organics (8260B)										
1,1,1,2-Tetrachloroethane	8260B	Water	ug/L	0.21	1	70	134	20		
1,1,1-Trichloroethane	8260B	Water	ug/L	0.22	1	66	129	20		
1,1,2,2-Tetrachloroethane	8260B	Water	ug/L	0.25	1	72	127	20		
1,1,2-Trichloroethane	8260B	Water	ug/L	0.33	1	69	138	20		
1,1-Dichloroethane	8260B	Water	ug/L	0.2	1	69	127	20		
1,1-Dichloroethene	8260B	Water	ug/L	0.19	1	54	127	20		
1,1-Dichloropropene	8260B	Water	ug/L	0.24	1	70	128	20		
1,2,3-Trichlorobenzene	8260B	Water	ug/L	0.24	1	75	123	20		

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
1,2,3-Trichloropropane	8260B	Water	ug/L	0.2	1	71	126	20		
1,2,4-Trichlorobenzene	8260B	Water	ug/L	0.23	1	77	123	20		
1,2,4-Trimethylbenzene	8260B	Water	ug/L	0.2	1	72	126	20		
1,2-Dibromo-3-chloropropane	8260B	Water	ug/L	0.46	1	66	123	20		
1,2-Dibromoethane (EDB)	8260B	Water	ug/L	0.25	1	71	135	20		
1,2-Dichlorobenzene	8260B	Water	ug/L	0.24	1	74	119	20		
1,2-Dichloroethane	8260B	Water	ug/L	0.25	1	63	133	20		
1,2-Dichloroethene (total)	8260B	Water	ug/L	0.42	1	72	121	20		
1,2-Dichloropropane	8260B	Water	ug/L	0.22	1	71	132	20		
1,3,5-Trichlorobenzene	8260B	Water	ug/L	0.24	1	72	127	20		
1,3,5-Trimethylbenzene	8260B	Water	ug/L	0.2	1	69	123	20		
1,3-Butadiene	8260B	Water	ug/L	0.25	1					
1,3-Dichlorobenzene	8260B	Water	ug/L	0.23	1	73	121	20		
1,3-Dichloropropane	8260B	Water	ug/L	0.23	1	71	133	20		
1,4-Dichlorobenzene	8260B	Water	ug/L	0.22	1	74	121	20		
1-Chlorohexane	8260B	Water	ug/L	0.23	1	71	139	20		
2,2-Dichloropropane	*8260B*	*Water*	*ug/L*	*0.2*	*1*	*56*	*141*	*20*		
2-Butanone (MEK)	8260B	Water	ug/L	1.7	5	54	145	20		
2-Chloro-1,3-butadiene (chloroprene)	8260B	Water	ug/L	0.13	1					
2-Chloroethylvinylether	8260B	Water	ug/L	1.4	2	10	200	20		
2-Chlorotoluene	8260B	Water	ug/L	0.22	1	69	120	20		
2-Hexanone	8260B	Water	ug/L	1.2	5	70	144	20		
2-Methylnaphthalene	8260B	Water	ug/L	0.27	1					
2-Nitropropane	8260B	Water	ug/L	20	100					
3-Chloropropene (Allyl Chloride)	8260B	Water	ug/L	0.45	2					
4-Chlorotoluene	8260B	Water	ug/L	0.22	1	68	120	20		
4-Methyl-2-pentanone (MIBK)	8260B	Water	ug/L	0.92	5	66	147	20		
Acetone	8260B	Water	ug/L	1.5	5	43	150	20		
Acetonitrile	8260B	Water	ug/L	6.6	40					
Acrolein	8260B	Water	ug/L	19	200					
Acrylonitrile	8260B	Water	ug/L	5.4	40					
Benzene	8260B	Water	ug/L	0.2	1	74	116	20		
bis(chloromethyl)ether	8260B	Water	ug/L	2000	2000					
Bromobenzene	8260B	Water	ug/L	0.22	1	77	121	20		
Bromochloromethane	8260B	Water	ug/L	0.19	1	57	133	20		
Bromodichloromethane	8260B	Water	ug/L	0.23	1	76	129	20		
Bromoform	8260B	Water	ug/L	0.22	1	73	139	20		
Bromomethane	8260B	Water	ug/L	0.18	1	51	152	20		

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Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Carbon disulfide	8260B	Water	ug/L	0.4	5	29	136	20		
Carbon tetrachloride	8260B	Water	ug/L	0.24	1	66	136	20		
Chlorobenzene	8260B	Water	ug/L	0.22	1	76	124	20		
Chloroethane	8260B	Water	ug/L	0.21	1	68	135	20		
Chloroform	8260B	Water	ug/L	0.23	1	74	128	20		
Chloromethane	8260B	Water	ug/L	0.16	1	56	129	20		
cis-1,2-Dichloroethene	8260B	Water	ug/L	0.21	1	78	126	20		
cis-1,3-Dichloropropene	8260B	Water	ug/L	0.22	1	75	123	20		
Cyclohexane	8260B	Water	ug/L	0.1	1					
Cyclohexanone	8260B	Water	ug/L	53	100					
Dibromochloromethane	8260B	Water	ug/L	0.23	1	74	137	20		
Dibromomethane	8260B	Water	ug/L	0.26	1	66	131	20		
Dichlorodifluoromethane	8260B	Water	ug/L	0.14	1	56	136	20		
Ethyl acetate	8260B	Water	ug/L	1.9	5					
Ethyl ether	8260B	Water	ug/L	0.31	1	10	190	20		
Ethylbenzene	8260B	Water	ug/L	0.2	1	74	121	20		
Ethylmethacrylate	8260B	Water	ug/L	0.36	2					
Heptane	8260B	Water	ug/L	0.22	1	50	143	20		
Hexachlorobutadiene	8260B	Water	ug/L	0.24	1	56	147	20		
Hexane	8260B	Water	ug/L	0.2	1	50	134	20		
Iodomethane	8260B	Water	ug/L	1.3	2	36	117	20		
Isobutyl alcohol	8260B	Water	ug/L	26	100					
Isopropyl alcohol	8260B	Water	ug/L							
Isopropyl ether	8260B	Water	ug/L	0.15	1					
Isopropylbenzene	8260B	Water	ug/L	0.21	1	67	123	20		
m&p-Xylenes	8260B	Water	ug/L	0.39	2	71	125	20		
Methacrylonitrile	8260B	Water	ug/L	1.5	2					
Methyl acetate	8260B	Water	ug/L	0.52	1					
Methyl cyclohexane	8260B	Water	ug/L	0.1	1					
Methylene chloride	8260B	Water	ug/L	0.19	1	52	133	20		
Methylmethacrylate	8260B	Water	ug/L	0.67	2					
Methyl-tert-butyl-ether (MTBE)	8260B	Water	ug/L	0.21	1	52	156	20		
Naphthalene	8260B	Water	ug/L	0.34	1	69	125	20		
n-Butyl alcohol (1-Butanol)	8260B	Water	ug/L	55	100					
n-Butylbenzene	8260B	Water	ug/L	0.22	1	71	118	20		
n-Pentane	8260B	Water	ug/L							
n-Propylbenzene	8260B	Water	ug/L	0.25	1	67	123	20		
o-Xylene	8260B	Water	ug/L	0.21	1	72	124	20		

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Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Pentachloroethane	8260B	Water	ug/L	1	2					
p-Isopropyltoluene	8260B	Water	ug/L	0.22	1	67	126	20		
Propionitrile	8260B	Water	ug/L	9.7	40					
sec-Butylbenzene	8260B	Water	ug/L	0.22	1	69	124	20		
Styrene	8260B	Water	ug/L	0.23	1	80	125	20		
tert-Butyl alcohol	8260B	Water	ug/L							
tert-Butylbenzene	8260B	Water	ug/L	0.21	1	69	123	20		
Tetrachloroethene	8260B	Water	ug/L	0.2	1	69	128	20		
Tetrahydrofuran	8260B	Water	ug/L	3	5	67	166	20		
Toluene	8260B	Water	ug/L	0.21	1	71	122	20		
trans-1,2-Dichloroethene	8260B	Water	ug/L	0.21	1	64	119	20		
trans-1,3-Dichloropropene	8260B	Water	ug/L	0.24	1	76	126	20		
trans-1,4-Dichloro-2-butene	8260B	Water	ug/L	1.3	2					
Trichloroethene	8260B	Water	ug/L	0.21	1	70	120	20		
Trichlorofluoromethane	8260B	Water	ug/L	0.22	1	62	141	20		
Trichlorotrifluoroethane	8260B	Water	ug/L	0.22	1	50	150	30		
Vinyl acetate	8260B	Water	ug/L	0.47	5	70	130	20		
Vinyl chloride	8260B	Water	ug/L	0.18	1	67	137	20		
Xylenes (total)	8260B	Water	ug/L	0.28	1	76	138	20		
Surrogate										
1,2-Dichloroethane-d4 (surr)	8260B	Water	ug/L						61	131
4-Bromofluorobenzene (surr)	8260B	Water	ug/L						73	122
Dibromofluoromethane (surr)	8260B	Water	ug/L						66	132
Toluene-d8 (surr)	8260B	Water	ug/L						78	128
Method: Volatile Organics (8260B)										
1,1,1,2-Tetrachloroethane	8260B	Solid	ug/Kg	0.73	5	83	123	20		
1,1,1-Trichloroethane	8260B	Solid	ug/Kg	0.61	5	63	133	20		
1,1,2,2-Tetrachloroethane	8260B	Solid	ug/Kg	0.64	5	68	139	20		
1,1,2-Trichloroethane	8260B	Solid	ug/Kg	0.71	5	71	143	20		
1,1-Dichloroethane	8260B	Solid	ug/Kg	0.88	5	63	133	20		
1,1-Dichloroethene	8260B	Solid	ug/Kg	1	5	51	132	20		
1,1-Dichloropropene	8260B	Solid	ug/Kg	0.8	5	78	148	20		
1,2,3-Trichlorobenzene	8260B	Solid	ug/Kg	0.99	5	75	125	20		
1,2,3-Trichloropropane	8260B	Solid	ug/Kg	1.1	5	71	129	20		
1,2,4-Trichlorobenzene	8260B	Solid	ug/Kg	0.79	5	76	127	20		
1,2,4-Trimethylbenzene	8260B	Solid	ug/Kg	0.82	5	74	133	20		
1,2-Dibromo-3-chloropropane	8260B	Solid	ug/Kg	1.1	5	59	124	20		
1,2-Dibromoethane (EDB)	8260B	Solid	ug/Kg	0.76	5	72	133	20		

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
1,2-Dichlorobenzene	8260B	Solid	ug/Kg	0.73	5	85	120	20		
1,2-Dichloroethane	8260B	Solid	ug/Kg	0.58	5	69	125	20		
1,2-Dichloroethene (total)	8260B	Solid	ug/Kg	1.9	5	63	144	20		
1,2-Dichloropropane	8260B	Solid	ug/Kg	0.96	5	76	132	20		
1,3,5-Trichlorobenzene	8260B	Solid	ug/Kg	1	5	70	131	20		
1,3,5-Trimethylbenzene	8260B	Solid	ug/Kg	0.58	5	72	128	20		
1,3-Butadiene	8260B	Solid	ug/Kg	0.93	5					
1,3-Dichlorobenzene	8260B	Solid	ug/Kg	0.91	5	83	122	20		
1,3-Dichloropropane	8260B	Solid	ug/Kg	0.93	5	78	127	20		
1,4-Dichlorobenzene	8260B	Solid	ug/Kg	0.89	5	84	121	20		
1-Chlorohexane	8260B	Solid	ug/Kg	1	5	62	145	20		
2,2-Dichloropropane	8260B	Solid	ug/Kg	1.3	5	67	134	20		
2-Butanone (MEK)	8260B	Solid	ug/Kg	4.2	5	50	150	30		
2-Chloro-1,3-butadiene (chloroprene)	8260B	Solid	ug/Kg	0.68	5					
2-Chloroethylvinylether	8260B	Solid	ug/Kg	0.82	5	10	182	20		
2-Chlorotoluene	8260B	Solid	ug/Kg	1	5	63	137	20		
2-Hexanone	8260B	Solid	ug/Kg	1.7	5	69	140	20		
2-Methylnaphthalene	8260B	Solid	ug/Kg	1.2	5					
2-Nitropropane	8260B	Solid	ug/Kg	139	400					
3-Chloropropene (Allyl Chloride)	8260B	Solid	ug/Kg	2.2	10					
4-Chlorotoluene	8260B	Solid	ug/Kg	0.77	5	76	123	20		
4-Methyl-2-pentanone (MIBK)	8260B	Solid	ug/Kg	3	5	68	134	20		
Acetone	8260B	Solid	ug/Kg	4.1	5	46	167	20		
Acetonitrile	8260B	Solid	ug/Kg	26	40					
Acrolein	8260B	Solid	ug/Kg	38	200					
Acrylonitrile	8260B	Solid	ug/Kg	7	40					
Benzene	8260B	Solid	ug/Kg	0.66	5	72	128	20		
bis(chloromethyl)ether	8260B	Solid	ug/Kg							
Bromobenzene	8260B	Solid	ug/Kg	0.71	5	81	123	20		
Bromochloromethane	8260B	Solid	ug/Kg	0.99	5	68	129	20		
Bromodichloromethane	8260B	Solid	ug/Kg	0.68	5	74	128	20		
Bromoform	8260B	Solid	ug/Kg	0.91	5	78	132	20		
Bromomethane	8260B	Solid	ug/Kg	2.9	5	48	127	20		
Carbon disulfide	8260B	Solid	ug/Kg	2	5	23	138	20		
Carbon tetrachloride	8260B	Solid	ug/Kg	0.83	5	67	127	20		
Chlorobenzene	8260B	Solid	ug/Kg	0.91	5	83	125	20		
Chloroethane	8260B	Solid	ug/Kg	1.6	5	59	163	20		
Chloroform	8260B	Solid	ug/Kg	0.62	5	73	135	20		

STL Chicago
Method Limit Report

Project:
Updated: 3/4/02

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Chloromethane	8260B	Solid	ug/Kg	0.94	5	45	141	20		
cis-1,2-Dichloroethene	8260B	Solid	ug/Kg	1.2	5	68	148	20		
cis-1,3-Dichloropropene	8260B	Solid	ug/Kg	0.79	5	80	124	20		
Cyclohexane	8260B	Solid	ug/Kg		5					
Cyclohexanone	8260B	Solid	ug/Kg	280	400					
Dibromochloromethane	8260B	Solid	ug/Kg	0.69	5	77	127	20		
Dibromomethane	8260B	Solid	ug/Kg	0.69	5	70	130	20		
Dichlorodifluoromethane	8260B	Solid	ug/Kg	0.75	5	43	121	20		
Ethyl acetate	8260B	Solid	ug/Kg	9.9	50					
Ethyl ether	8260B	Solid	ug/Kg	1.2	5	50	150	30		
Ethylbenzene	8260B	Solid	ug/Kg	1.1	5	79	123	20		
Ethylmethacrylate	8260B	Solid	ug/Kg	1.5	10					
Heptane	8260B	Solid	ug/Kg	0.75	5	50	150	30		
Hexachlorobutadiene	8260B	Solid	ug/Kg	1	5	66	127	20		
Hexane	8260B	Solid	ug/Kg	0.61	5	50	150	30		
Iodomethane	8260B	Solid	ug/Kg	3.5	10	50	150	30		
Isobutyl alcohol	8260B	Solid	ug/Kg	84	400					
Isopropyl alcohol	8260B	Solid	ug/Kg							
Isopropyl ether	8260B	Solid	ug/Kg	0.64	5					
Isopropylbenzene	8260B	Solid	ug/Kg	0.75	5	77	118	20		
m&p-Xylenes	8260B	Solid	ug/Kg	2.1	10	79	123	20		
Methacrylonitrile	8260B	Solid	ug/Kg	4.3	10					
Methyl acetate	8260B	Solid	ug/Kg		5					
Methyl cyclohexane	8260B	Solid	ug/Kg		5					
Methylene chloride	8260B	Solid	ug/Kg	1.8	5	58	143	20		
Methylmethacrylate	8260B	Solid	ug/Kg	2	10					
Methyl-tert-butyl-ether (MTBE)	8260B	Solid	ug/Kg	0.64	5	61	132	20		
Naphthalene	8260B	Solid	ug/Kg	1	5	65	132	20		
n-Butyl alcohol (1-Butanol)	8260B	Solid	ug/Kg	178	400					
n-Butylbenzene	8260B	Solid	ug/Kg	0.84	5	65	138	20		
n-Pentane	8260B	Solid	ug/Kg							
n-Propylbenzene	8260B	Solid	ug/Kg	0.86	5	77	124	20		
o-Xylene	8260B	Solid	ug/Kg	0.93	5	80	123	20		
Pentachloroethane	8260B	Solid	ug/Kg	5.4	10					
p-Isopropyltoluene	8260B	Solid	ug/Kg	0.68	5	74	126	20		
Propionitrile	8260B	Solid	ug/Kg	19	40					
sec-Butylbenzene	8260B	Solid	ug/Kg	0.81	5	77	128	20		
Styrene	8260B	Solid	ug/Kg	1	5	85	126	20		

STL Chicago
Method Limit Report

Project:
Updated: 3/4/02

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
tert-Butyl alcohol	8260B	Solid	ug/Kg							
tert-Butylbenzene	8260B	Solid	ug/Kg	0.78	5	79	124	20		
Tetrachloroethene	8260B	Solid	ug/Kg	0.67	5	75	129	20		
Tetrahydrofuran	8260B	Solid	ug/Kg	2.5	5	55	135	20		
Toluene	8260B	Solid	ug/Kg	1	5	75	125	20		
trans-1,2-Dichloroethene	8260B	Solid	ug/Kg	0.94	5	58	139	20		
trans-1,3-Dichloropropene	8260B	Solid	ug/Kg	0.84	5	75	134	20		
trans-1,4-Dichloro-2-butene	8260B	Solid	ug/Kg	2.2	10					
Trichloroethene	8260B	Solid	ug/Kg	0.59	5	75	129	20		
Trichlorofluoromethane	8260B	Solid	ug/Kg	0.71	5	57	135	20		
Trichlorotrifluoroethane	8260B	Solid	ug/Kg	1.8	5	50	150	30		
Vinyl acetate	8260B	Solid	ug/Kg	0.56	5	50	150	30		
Vinyl chloride	8260B	Solid	ug/Kg	0.74	5	58	140	20		
Xylenes (total)	8260B	Solid	ug/Kg	2.9	5	82	125	20		
Surrogate										
1,2-Dichloroethane-d4 (surr)	8260B	Solid	ug/Kg						50	145
4-Bromofluorobenzene (surr)	8260B	Solid	ug/Kg						60	140
Dibromofluoromethane (surr)	8260B	Solid	ug/Kg						60	140
Toluene-d8 (surr)	8260B	Solid	ug/Kg						66	141
Method: Volatile Organics (8260B)										
1,1,1,2-Tetrachloroethane	8260B	High/MeOH	ug/Kg	25.5	100	74	120	30		
1,1,1-Trichloroethane	8260B	High/MeOH	ug/Kg	16.5	100	69	133	30		
1,1,2,2-Tetrachloroethane	8260B	High/MeOH	ug/Kg	18.5	100	70	126	30		
1,1,2-Trichloroethane	8260B	High/MeOH	ug/Kg	31.5	100	67	133	30		
1,1-Dichloroethane	8260B	High/MeOH	ug/Kg	13.5	100	68	119	30		
1,1-Dichloroethene	8260B	High/MeOH	ug/Kg	14	100	44	143	30		
1,1-Dichloropropene	8260B	High/MeOH	ug/Kg	18.5	100	65	134	30		
1,2,3-Trichlorobenzene	8260B	High/MeOH	ug/Kg	49	100	68	117	30		
1,2,3-Trichloropropane	8260B	High/MeOH	ug/Kg	49	100	64	118	30		
1,2,4-Trichlorobenzene	8260B	High/MeOH	ug/Kg	41.5	100	61	117	30		
1,2,4-Trimethylbenzene	8260B	High/MeOH	ug/Kg	23	100	69	122	30		
1,2-Dibromo-3-chloropropane	8260B	High/MeOH	ug/Kg	22.5	100	56	102	30		
1,2-Dibromoethane (EDB)	8260B	High/MeOH	ug/Kg	25.5	100	69	122	30		
1,2-Dichlorobenzene	8260B	High/MeOH	ug/Kg	17	100	76	125	30		
1,2-Dichloroethane	8260B	High/MeOH	ug/Kg	21.5	100	64	115	30		
1,2-Dichloroethene (total)	8260B	High/MeOH	ug/Kg	29	100	60	139	30		
1,2-Dichloropropane	8260B	High/MeOH	ug/Kg	17.5	100	70	122	30		
1,3,5-Trichlorobenzene	8260B	High/MeOH	ug/Kg	29	100	65	113	30		

STL Chicago
Method Limit Report

Project:
Updated: 3/4/02

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
1,3,5-Trimethylbenzene	8260B	High/MeOH	ug/Kg	19.5	100	66	125	30		
1,3-Butadiene	8260B	High/MeOH	ug/Kg		100					
1,3-Dichlorobenzene	8260B	High/MeOH	ug/Kg	23	100	75	119	30		
1,3-Dichloropropane	8260B	High/MeOH	ug/Kg	23.5	100	71	118	30		
1,4-Dichlorobenzene	8260B	High/MeOH	ug/Kg	20.5	100	76	127	30		
1-Chlorohexane	8260B	High/MeOH	ug/Kg	22.5	100	63	133	30		
2,2-Dichloropropane	8260B	High/MeOH	ug/Kg	11.5	100	41	131	30		
2-Butanone (MEK)	8260B	High/MeOH	ug/Kg	51	100	40	125	30		
2-Chloro-1,3-butadiene (chloroprene)	8260B	High/MeOH	ug/Kg		100					
2-Chloroethylvinylether	8260B	High/MeOH	ug/Kg	76	100	10	100	30		
2-Chlorotoluene	8260B	High/MeOH	ug/Kg	40.5	100	62	134	30		
2-Hexanone	8260B	High/MeOH	ug/Kg	52	100	50	116	30		
2-Methylnaphthalene	8260B	High/MeOH	ug/Kg		100					
2-Nitropropane	8260B	High/MeOH	ug/Kg		8000					
3-Chloropropene (Allyl Chloride)	8260B	High/MeOH	ug/Kg	17	200					
4-Chlorotoluene	8260B	High/MeOH	ug/Kg	23	100	66	131	30		
4-Methyl-2-pentanone (MIBK)	8260B	High/MeOH	ug/Kg	37.5	100	54	119	30		
Acetone	8260B	High/MeOH	ug/Kg	29	100	34	143	30		
Acetonitrile	8260B	High/MeOH	ug/Kg	522	800					
Acrolein	8260B	High/MeOH	ug/Kg	1473	4000					
Acrylonitrile	8260B	High/MeOH	ug/Kg	307	800					
Benzene	8260B	High/MeOH	ug/Kg	14	100	67	122	30		
bis(chloromethyl)ether	8260B	High/MeOH	ug/Kg							
Bromobenzene	8260B	High/MeOH	ug/Kg	27.5	100	74	133	30		
Bromochloromethane	8260B	High/MeOH	ug/Kg	24.5	100	60	124	30		
Bromodichloromethane	8260B	High/MeOH	ug/Kg	19	100	66	128	30		
Bromoform	8260B	High/MeOH	ug/Kg	18	100	70	123	30		
Bromomethane	8260B	High/MeOH	ug/Kg	10.5	100	36	164	30		
Carbon disulfide	8260B	High/MeOH	ug/Kg	20.5	100	21	124	30		
Carbon tetrachloride	8260B	High/MeOH	ug/Kg	16.5	100	59	127	30		
Chlorobenzene	8260B	High/MeOH	ug/Kg	22	100	80	125	30		
Chloroethane	8260B	High/MeOH	ug/Kg	20	100	33	207	30		
Chloroform	8260B	High/MeOH	ug/Kg	18	100	61	129	30		
Chloromethane	8260B	High/MeOH	ug/Kg	23.5	100	55	129	30		
cis-1,2-Dichloroethene	8260B	High/MeOH	ug/Kg	17	100	64	144	30		
cis-1,3-Dichloropropene	8260B	High/MeOH	ug/Kg	22.5	100	68	123	30		
Cyclohexane	8260B	High/MeOH	ug/Kg		100					
Cyclohexanone	8260B	High/MeOH	ug/Kg		8000					

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
Dibromochloromethane	8260B	High/MeOH	ug/Kg	19	100	70	119	30		
Dibromomethane	8260B	High/MeOH	ug/Kg	22.5	100	67	121	30		
Dichlorodifluoromethane	8260B	High/MeOH	ug/Kg	12	100	29	135	30		
Ethyl acetate	8260B	High/MeOH	ug/Kg	108	1000					
Ethyl ether	8260B	High/MeOH	ug/Kg	19	100	10	135	30		
Ethylbenzene	8260B	High/MeOH	ug/Kg	22.5	100	78	128	30		
Ethylmethacrylate	8260B	High/MeOH	ug/Kg	24	200					
Heptane	8260B	High/MeOH	ug/Kg	38	100	10	119	30		
Hexachlorobutadiene	8260B	High/MeOH	ug/Kg	38.5	100	63	126	30		
Hexane	8260B	High/MeOH	ug/Kg	27.5	100	10	108	30		
Iodomethane	8260B	High/MeOH	ug/Kg	25.5	200	50	150	30		
Isobutyl alcohol	8260B	High/MeOH	ug/Kg	3740	8000					
Isopropyl alcohol	8260B	High/MeOH	ug/Kg							
Isopropyl ether	8260B	High/MeOH	ug/Kg	9.5	100					
Isopropylbenzene	8260B	High/MeOH	ug/Kg	20	100	67	133	30		
m&p-Xylenes	8260B	High/MeOH	ug/Kg	50	200	76	133	30		
Methacrylonitrile	8260B	High/MeOH	ug/Kg	40.5	200					
Methyl acetate	8260B	High/MeOH	ug/Kg		100					
Methyl cyclohexane	8260B	High/MeOH	ug/Kg		100					
Methylene chloride	8260B	High/MeOH	ug/Kg	20	100	57	129	30		
Methylmethacrylate	8260B	High/MeOH	ug/Kg	19	200					
Methyl-tert-butyl-ether (MTBE)	8260B	High/MeOH	ug/Kg	30.5	100	47	126	30		
Naphthalene	8260B	High/MeOH	ug/Kg	38	100	51	158	30		
n-Butyl alcohol (1-Butanol)	8260B	High/MeOH	ug/Kg	8000	8000					
n-Butylbenzene	8260B	High/MeOH	ug/Kg	18.5	100	64	118	30		
n-Pentane	8260B	High/MeOH	ug/Kg							
n-Propylbenzene	8260B	High/MeOH	ug/Kg	27.5	100	69	130	30		
o-Xylene	8260B	High/MeOH	ug/Kg	23.5	100	74	127	30		
Pentachloroethane	8260B	High/MeOH	ug/Kg	83	200					
p-Isopropyltoluene	8260B	High/MeOH	ug/Kg	23.5	100	68	129	30		
Propionitrile	8260B	High/MeOH	ug/Kg	360	800					
sec-Butylbenzene	8260B	High/MeOH	ug/Kg	20.5	100	69	139	30		
Styrene	8260B	High/MeOH	ug/Kg	28.5	100	80	129	30		
tert-Butyl alcohol	8260B	High/MeOH	ug/Kg							
tert-Butylbenzene	8260B	High/MeOH	ug/Kg	13.5	100	71	125	30		
Tetrachloroethene	8260B	High/MeOH	ug/Kg	23	100	75	125	30		
Tetrahydrofuran	8260B	High/MeOH	ug/Kg	43	100	36	132	30		
Toluene	8260B	High/MeOH	ug/Kg	18	100	72	123	30		

Method Description	Analytical Method	Test Matrix	Units	MDL	Lab RL	LCL	UCL	RPD	SLL	SUL
trans-1,2-Dichloroethene	8260B	High/MeOH	ug/Kg	13.5	100	66	138	30		
trans-1,3-Dichloropropene	8260B	High/MeOH	ug/Kg	19.5	100	60	115	30		
trans-1,4-Dichloro-2-butene	8260B	High/MeOH	ug/Kg	30.5	200					
Trichloroethene	8260B	High/MeOH	ug/Kg	21.5	100	70	123	30		
Trichlorofluoromethane	8260B	High/MeOH	ug/Kg	19.5	100	59	145	30		
Trichlorotrifluoroethane	8260B	High/MeOH	ug/Kg	15	100	50	150	30		
Vinyl acetate	8260B	High/MeOH	ug/Kg	32	100	54	144	30		
Vinyl chloride	8260B	High/MeOH	ug/Kg	18	100	61	135	30		
Xylenes (total)	8260B	High/MeOH	ug/Kg	50	100	77	131	30		
Surrogate										
1,2-Dichloroethane-d4 (surr)	8260B	High/MeOH	ug/Kg						43	139
4-Bromofluorobenzene (surr)	8260B	High/MeOH	ug/Kg						57	124
Dibromofluoromethane (surr)	8260B	High/MeOH	ug/Kg						64	132
Toluene-d8 (surr)	8260B	High/MeOH	ug/Kg						70	128

Notes:

MDLs will vary based on annual performance.

RLs will vary based on sample volume/size; dilution factors; dry weight reporting (soils) and annual MDL determinations.

Table 2

Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion
Benzene	78	—
Bromobenzene	156	77,158
Bromochloromethane	128	49,130
Bromodichloromethane	83	85,127
Bromoform	173	175,254
Bromomethane	94	96
n-Butylbenzene	91	92,134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91,134
Carbon tetrachloride	117	119
Chlorobenzene	112	77,114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155,157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Dibromomethane	93	95,174
1,2-Dichlorobenzene	146	111,148
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61,63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110,77
Ethylbenzene	91	106
Hexachlorobutadiene	225	223,227
Isopropylbenzene	105	120
p-Isopropylbenzene	119	134, 91
Methylene chloride	84	86,49
Naphthalene	128	86, 49
n-Propylbenzene	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129,131,166

Table 2
(continued)
Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101,153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
Internal Std./Surrogates		
4-Bromofluorobenzene (S)	95	174, 176
1,4-Dichlorobenzene-d_4 (IS)	152	115, 150
Pentafluorobenzene (IS)	168	
Chlorobenzene-d_5 (IS)	117	
1,4-Difluorobenzene (IS)	114	
1,2-Dichloroethane-d_4 (S)	65	
Toluene-d_8 (S)	98	
Dibromofluoromethane (S)	113	

*NOTE: The primary and secondary ions listed here are taken directly from SW-846 Method 8260. The laboratory uses secondary ions in the cases of Ethylbenzene, Toluene, 1,1,2-Trichloroethane, Trichloroethene, 1,2,3-Trichloropropane and Xylenes due to interferences and/or to maintain consistency between methods.

Figure 1.

Example: Total Ion Chromatogram for 25 mL Purge Water

Data File: /var/chem/gc13.i/101802.b/3c1017.d
Date : 18-OCT-2002 08:19
Client ID: VSTD010
Sample Info: CV V02J18DAA
Purge Volume: 25.0
Column phase: Cap

Instrument: gc13.i
Operator: DCT
Column diameter: 0.53

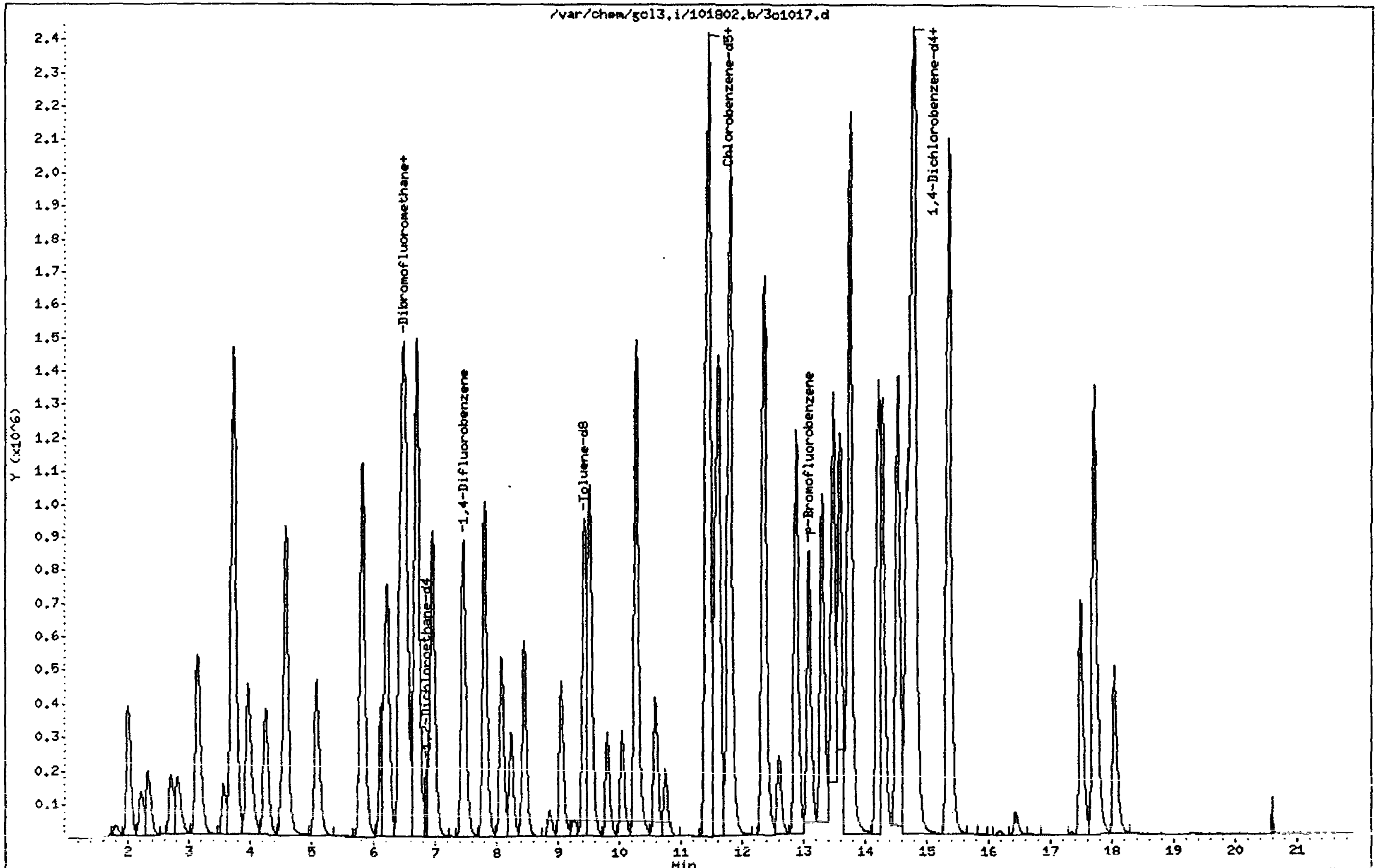


Figure 2.

Example: Total Ion Chromatogram for 5 mL Purge Soil

Data File: /var/chem/gc19.i/090602_5ml9w.b/9i0906n.d

Date : 06-SEP-2002 06:25

Client ID: VSTD050

Instrument: gc19.i

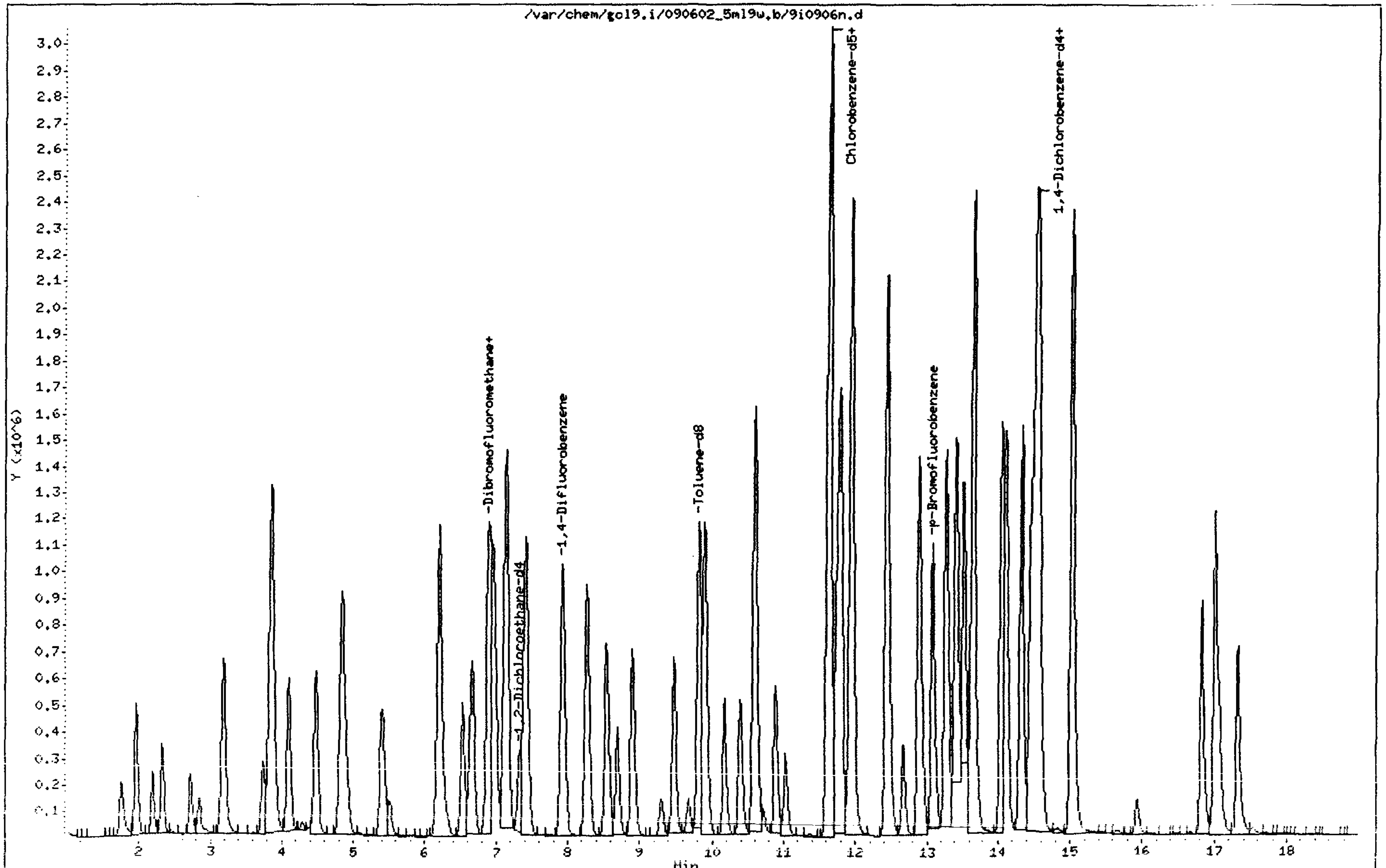
Sample Info: VSTD050

Purge Volume: 5.0

Operator: LM

Column phase: Cap

Column diameter: 0.53



Attachment 1.

Example: Method Listings; Tekmar Conditions; Flow Settings

Example: Volatiles Method for Standards and Samples

GC Oven Parameters

Initial Temperature = 40 °C
Initial Time = 2.0 minutes
Detector A Temperature = 180 °C
Detector B Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
7.0	65	0.00
12.0	165	0.00
20.0	212	5.00

Run Time = 21.25 min.

Inlet Pressure Program

Gas = Helium
Column length = 75 m
Column Diameter = 0.530 mm
Initial Pressure = 3 psi
Rate (psi/min) = 0.00
Initial Time = 7.0 min.
Oven Temp. 50 °C
Program Time = 7.0 min.

Scan Parameters

Mass Range = 35-260
Threshold = 150
Scans/sec = 1.9
EM Voltage = 1938
Solvent Delay = 3.14 (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Example: Volatiles Method for BFB Tune

GC Oven Parameters

Initial Temperature = 40 °C
Initial Time = 2.0 minutes
Detector A Temperature = 180 °C
Detector B Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
7.0	65	0.00
12.0	165	0.00
20.0	212	5.00

Run Time = 16.25 min.

Inlet Pressure Program

Gas = Helium
Column length = 75 m
Column Diameter = 0.530 mm
Initial Pressure = 3 psi
Rate (psi/min) = 0.00
Initial Time = 7.0 min.
Oven Temp. 50 °C
Program Time = 7.0 min.

Scan Parameters

Mass Range = 35-260
Threshold = 150
Scans/sec = 1.9
EM Voltage = 1938
Solvent Delay = 10 min. (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Tekmar Conditions

Trap Temp. Prior to Purge	< 35
Desorb Preheat	250
Desorb	250
Bake	260
Purge Time	11 *min*
Desorb	2 min
Bake Time	4 min

Trap = Vocarb 3000

Flow Conditions

Purge Pressure	*20 psi*
Purge Flow Rate	~40 mLs/min

Flow Adjustment

Capillary Column: 5971/5972/MSD's; · Make-up gas off/separator pump on: flow through separator *is 5-10 mLs/minutes.* · Open make-up gas: adjust until you achieve ~30 mLs/minute through the separator. (On MSD's - adjust to 0.5 torr on gauge) (Flow into the Mass Spec is ≤ 1 mL/minute)

Approximate Vacuums

5971	~5 x 10^{-6} torr
5972	~5 x 10^{-6} torr

Attachment 2.

**Example: Target and Internal Standards;
Initial Calibration/Continuing Calibration
Surrogate Recovery Limits; LCS / MS Recovery Limits
Internal Standard Guidelines**

Target and Internal Standards

Pentafluorobenzene
Acetone
Acrolein
Acrylonitrile
Bromochloromethane
Bromomethane
2-Butanone
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
Dichlorodifluoromethane
1,1-Dichloroethane
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
2,2-Dichloropropane
Iodomethane
Methylene chloride
1,1,1-Trichloroethane
Trichlorofluoromethane
Vinyl acetate
Vinyl Chloride

Chlorobenzene-d_5
Bromoform
Bromofluorobenzene (surrogate)
Chlorodibromomethane
Chlorobenzene
1,3-Dichloropropane
Ethylbenzene
2-Hexanone
Styrene
1 1,1,2-Tetrachloroethane
Tetrachloroethene
Xylene

1,4-Difluorobenzene
Benzene
Bromodichloromethane
Carbon tetrachloride
2-Chloroethyl vinyl ether
1,2-Dibromoethane
Dibromomethane
1,2-Dichloroethane
1,2-Dichloroethane-d_4 (surrogate)
1,2-Dichloropropane
1,1-Dichloropropene
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
4-Methyl-2-pentanone
Toluene
Toluene-d_8 (surrogate)
1,1,2-Trichloroethane
Trichloroethene

1,4-Dichlorobenzene-d_4
Bromobenzene
n-Butylbenzene
sec-Butylbenzene
tert-Butylbenzene
2-Chlorotoluene
4-Chlorotoluene
1,2-Dibromo-3-chloropropane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Hexachlorobutadiene
Isopropyl benzene
p-Isopropyltoluene
Naphthalene
n-Propylbenzene
1,1,2,2-Tetrachloroethane
1,2,3-Trichlorobenzene
1,2,4-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

INITIAL CALIBRATION DATA

Start Cal Date : 03-SEP-2002 16:36
End Cal Date : 04-SEP-2002 05:13
Quant Method : ISTD
Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gcl6.i/090402_25ml6w_ical2.b/25ml6w.m
Cal Date : 20-Sep-2002 11:40 beeson

Calibration File Names:
Level 1: /var/chem/gcl6.i/090402_25ml6w_ical2.b/6i0904.d
Level 2: /var/chem/gcl6.i/090402_25ml6w_ical2.b/6i0904a.d
Level 3: /var/chem/gcl6.i/090402_25ml6w_ical2.b/6i0904b.d
Level 4: /var/chem/gcl6.i/090402_25ml6w_ical2.b/6i0904c.d
Level 5: /var/chem/gcl6.i/090402_25ml6w_ical2.b/6i0904d.d
Level 6: /var/chem/gcl6.i/090402_25ml6w_ical2.b/6i0904e.d
Level 7: /var/chem/gcl6.i/090402_25ml6w_ical2.b/6i0904f.d
Level 8: /var/chem/gcl6.i/090402_25ml6w_ical2.b/6i0904g.d
Level 9: /var/chem/gcl6.i/090402_25ml6w_ical2.b/6i0904h.d

Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
1 Dichlorodifluoromethane	39157	71069	139450	346832	800413	1070808					
	1337714	2115698	4000493				LINR	0.04663	0.52169		0.99787
2 Chloromethane	0.33705	0.30726	0.28187	0.26440	0.28090	0.27369					
	0.27310	0.27412	0.27691				AVRG		0.28548		7.93729
3 2-Propanol	+++++	+++++	+++++	+++++	+++++	+++++					
	+++++	+++++	+++++				AVRG		0.000e+00		0.000e+00 <-
4 Vinyl chloride	0.33399	0.33547	0.32629	0.32674	0.33838	0.35039					
	0.34067	0.34890	0.35023				AVRG		0.33901		2.77218
5 1,3-Butadiene	+++++	0.19071	0.16517	0.15550	0.17524	0.17008					
	0.16454	0.16167	0.15891				AVRG		0.16773		6.65034

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Cal Date : 20-Sep-2002 11:40 beeson

Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
6 Bromomethane	0.23835	0.23980	0.17896	0.20284	0.22226	0.23954					
	0.25249	0.25844	0.29868				AVRG		0.23682		14.37674
7 Chloroethane	0.21870	0.21455	0.21124	0.20788	0.20578	0.20705					
	0.20279	0.20547	0.21123				AVRG		0.20941		2.38673
8 tert-Butyl alcohol	+++++	+++++	+++++	+++++	+++++	+++++					
	+++++	+++++	+++++				AVRG		0.000e+00		0.000e+00 <-
9 Trichlorofluoromethane	0.83703	0.81527	0.78708	0.78714	0.75862	0.79943					
	0.74508	0.79775	0.78188				AVRG		0.78992		3.49126
10 Ethyl ether	+++++	0.08362	0.07579	0.07203	0.08150	0.07721					
	0.07591	0.07506	0.07547				AVRG		0.07708		4.84254
11 Acrolein	+++++	0.00421	0.00411	0.00405	0.00398	0.00415					
	0.00435	0.00405	0.00427				AVRG		0.00414		3.00288
12 Trichlorotrifluoroethane	0.76959	0.75887	0.71293	0.72786	0.73643	0.73957					
	0.76697	0.73146	0.75768				AVRG		0.74460		2.62127
13 1,1-Dichloroethene	0.33191	0.34084	0.31363	0.32090	0.32449	0.31844					
	0.33942	0.32259	0.33517				AVRG		0.32749		2.95473
14 Acetone	+++++	9564	21917	23823	32764	40741					
	48500	64187	124629				LINR	-0.27751	0.01485		0.99517

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Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
15 Iodomethane	+++++	0.48722	0.52068	0.66488	0.67114	0.71473					
	0.70443	0.70106	0.73275				AVRG		0.64961		14.31034
16 Carbon Disulfide	+++++	1.04025	1.04085	1.03106	1.01126	1.04452					
	1.08102	1.06338	1.11121				AVRG		1.05294		2.97886
17 3-Chloropropene	+++++	0.13576	0.13166	0.14736	0.13622	0.13873					
	0.13252	0.13223	0.13263				AVRG		0.13589		3.86439
18 Acetonitrile	+++++	0.00333	0.00306	0.00302	0.00292	0.00301					
	0.00313	0.00293	0.00317				AVRG		0.00307		4.45171
19 Methyl acetate	+++++	0.04115	0.04185	0.03764	0.03939	0.03823					
	0.03828	0.03689	0.03641				AVRG		0.03873		5.01965
20 Methylene chloride	61630	83247	134095	257213	384502	480778					
	657833	890236	1804838				LINR	-0.07949	0.22395		0.99901
21 Acrylonitrile	+++++	0.01252	0.01237	0.01190	0.01165	0.01193					
	0.01289	0.01209	0.01304				AVRG		0.01230		4.02194
22 trans-1,2-Dichloroethene	0.37667	0.37477	0.36652	0.37254	0.36192	0.36342					
	0.38164	0.36427	0.38282				AVRG		0.37162		2.13406
23 Methyl-tert-Butyl Ether	0.19519	0.19754	0.18760	0.19308	0.17962	0.18467					
	0.18447	0.18778	0.20188				AVRG		0.19020		3.77049

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Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	Coefficients m1	Coefficients m2	%RSD or R^2
24 Hexane	+++++	0.47742	0.45314	0.44029	0.49657	0.47793					
	0.46598	0.46287	0.46209				AVRG		0.46704		3.66451
25 1,1-Dichloroethane	0.63074	0.65386	0.64187	0.64979	0.62827	0.61320					
	0.65432	0.62062	0.65801				AVRG		0.63896		2.55271
26 Isopropyl ether	+++++	0.69384	0.66336	0.63824	0.72288	0.69174					
	0.67710	0.67794	0.68170				AVRG		0.68085		3.59695
27 Vinyl Acetate	+++++	0.10934	0.10342	0.10442	0.09682	0.10672					
	0.11132	0.11140	0.11356				AVRG		0.10713		5.10756
28 2-Chloro-1,3-butadiene	+++++	0.49643	0.48947	0.47927	0.54544	0.52427					
	0.52092	0.51603	0.51800				AVRG		0.51123		4.19320
29 cis-1,2-Dichloroethene	0.33006	0.31885	0.31590	0.32003	0.31505	0.30871					
	0.32918	0.31067	0.32844				AVRG		0.31966		2.50455
30 2,2-Dichloropropane	0.55090	0.55456	0.53095	0.54062	0.52930	0.51855					
	0.54667	0.51410	0.53199				AVRG		0.53529		2.61558
31 2-Butanone	+++++	0.02741	0.02302	0.02200	0.02132	0.02174					
	0.02167	0.02058	0.02114				AVRG		0.02236		9.66688
32 Ethyl Acetate	0.05503	0.05755	0.05476	0.05905	0.05625	0.05647					
	0.05804	0.05488	0.04197				AVRG		0.05600		10.88497

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Cal Date : 20-Sep-2002 11:40 beeson

Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	Coefficients m1	Coefficients m2	%RSD or R^2
33 Propionitrile	+++++	0.00332	0.00329	0.00327	0.00329	0.00338					
	0.00356	0.00336	0.00349				AVRG		0.00337		3.08896
34 Methacrylonitrile	+++++	0.03048	0.02925	0.03130	0.02931	0.03026					
	0.02805	0.02835	0.02859				AVRG		0.02945		3.87174
35 Bromochloromethane	0.14712	0.14945	0.14583	0.15098	0.14811	0.14596					
	0.15315	0.14711	0.15547				AVRG		0.14924		2.24709
36 Tetrahydrofuran	0.01458	0.01290	0.01215	0.01143	0.01127	0.01126					
	0.01128	0.01133	0.01236				AVRG		0.01206		9.24601
37 Chloroform	0.59355	0.59702	0.60304	0.61248	0.60630	0.58623					
	0.62611	0.58572	0.61988				AVRG		0.60337		2.36083
40 1,1,1-Trichloroethane	0.63602	0.63397	0.61618	0.62727	0.61453	0.60379					
	0.63444	0.61039	0.63361				AVRG		0.62336		1.96215
41 Cyclohexane	+++++	0.61584	0.56015	0.55126	0.63389	0.60884					
	0.58981	0.57444	0.57430				AVRG		0.58856		4.89600
M 42 1,2-Dichloroethene (total)	0.35336	0.34681	0.34121	0.34629	0.33849	0.33607					
	0.35541	0.33747	0.35563				AVRG		0.34564		2.25460
43 1,1-Dichloropropene	0.58998	0.56290	0.54002	0.55507	0.53943	0.54123					
	0.56771	0.53644	0.56076				AVRG		0.55484		3.17416

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44 Carbon tetrachloride	0.49844	0.51861	0.52698	0.53588	0.54359	0.54050						
	0.56030	0.54585	0.55748				AVRG		0.53640		3.62204	
45 Isobutanol	+++++	51678	86762	159054	246620	268229						
	395231	552369	855921				LINR	-4.33380	0.00138		0.99450	
47 Benzene	0.70232	0.68295	0.68810	0.67813	0.68193	0.68489						
	0.70878	0.67154	0.69919				AVRG		0.68865		1.77758	
48 1,2-Dichloroethane	0.15378	0.15130	0.15211	0.15301	0.15256	0.14861						
	0.15326	0.14900	0.15233				AVRG		0.15177		1.20527	
49 Heptane	+++++	0.45805	0.45648	0.42780	0.51395	0.49157						
	0.47962	0.47516	0.46437				AVRG		0.47087		5.47216	
51 Crotononitrile	+++++	+++++	+++++	+++++	+++++	+++++						
	+++++	+++++	+++++				AVRG		0.000e+00		0.000e+00	<-
52 n-Butanol	+++++	0.00084	0.00090	0.00089	0.00091	0.00088						
	0.00082	0.00088	0.00091				AVRG		0.00088		3.61232	<-
53 Trichloroethene	0.43224	0.41530	0.41755	0.42077	0.43010	0.42709						
	0.44027	0.42384	0.43293				AVRG		0.42668		1.89372	
54 Methyl cyclohexane	+++++	0.43094	0.42949	0.42248	0.48429	0.47589						
	0.45846	0.45464	0.44922				AVRG		0.45067		4.94594	

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Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	Coefficients m1	Coefficients m2	%RSD or R^2
55 1,2-Dichloropropane	0.26763	0.25355	0.24897	0.25365	0.26017	0.25860					
	0.27352	0.25464	0.26446				AVRG		0.25946		3.02396
56 Methylmethacrylate	+++++	0.04897	0.05007	0.05367	0.05107	0.05352					
	0.05123	0.05219	0.05260				AVRG		0.05166		3.18548
57 Dibromomethane	0.13296	0.13502	0.13056	0.13674	0.14013	0.13845					
	0.14346	0.13463	0.14008				AVRG		0.13689		2.94960
58 Bromodichloromethane	0.35060	0.35932	0.35813	0.38080	0.40075	0.39491					
	0.41350	0.38745	0.40545				AVRG		0.38343		5.93503
59 2-Nitropropane	+++++	0.00745	0.00808	0.01015	0.01038	0.01113					
	0.01076	0.01107	0.01118				AVRG		0.01002		14.48459 <-
60 2-Chloroethylvinylether	+++++	0.05348	0.05546	0.05265	0.05730	0.05820					
	0.05841	0.05675	0.05895				AVRG		0.05640		4.14094
61 cis-1,3-Dichloropropene	0.27279	0.26454	0.27235	0.27242	0.29143	0.28507					
	0.29986	0.27833	0.29180				AVRG		0.28095		4.15805
62 4-Methyl-2-pentanone	+++++	0.04393	0.04801	0.04592	0.04716	0.05058					
	0.05127	0.04803	0.04879				AVRG		0.04796		4.95596
64 Toluene	0.51253	0.48385	0.47090	0.48241	0.49247	0.48724					
	0.51041	0.47512	0.49422				AVRG		0.48991		2.91980

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65 trans-1,3-Dichloropropene	0.16082	0.16338	0.16087	0.16413	0.17941	0.17579					
	0.18413	0.17201	0.18008				AVRG		0.17118		5.30878
66 Bis(chloromethyl)ether	+++++	+++++	+++++	+++++	+++++	+++++					
	+++++	+++++	+++++				AVRG		0.000e+00		0.000e+00 <-
67 Ethylmethacrylate	+++++	0.13810	0.13752	0.15459	0.14804	0.14974					
	0.14641	0.14517	0.14867				AVRG		0.14603		3.96212
68 1,1,2-Trichloroethane	0.13251	0.13561	0.13344	0.13623	0.14149	0.13817					
	0.14394	0.13181	0.13924				AVRG		0.13694		3.02214
69 Tetrachloroethene	0.64754	0.61450	0.61012	0.60808	0.63078	0.64273					
	0.65249	0.61841	0.62254				AVRG		0.62747		2.65728
70 1,3-Dichloropropane	0.27671	0.26638	0.26184	0.26338	0.27963	0.27891					
	0.28467	0.26755	0.27006				AVRG		0.27212		2.96658
71 2-Hexanone	+++++	0.03946	0.03984	0.04169	0.04336	0.04725					
	0.04388	0.04405	0.04406				AVRG		0.04295		5.92456
72 Dibromochloromethane	0.32972	0.33044	0.33807	0.36214	0.39859	0.39047					
	0.40848	0.39372	0.39643				AVRG		0.37201		8.61171
73 1,2-Dibromoethane	0.17099	0.16916	0.16973	0.17168	0.18323	0.18300					
	0.18819	0.17580	0.18172				AVRG		0.17706		4.00533

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Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
74 1-Chlorohexane	0.54281	0.50632	0.48831	0.48792	0.46229	0.47937					
	0.47406	0.49425	0.53410				AVRG		0.49660		5.40703
76 Chlorobenzene	0.91801	0.84232	0.83922	0.85452	0.90675	0.89054					
	0.92204	0.86387	0.87376				AVRG		0.87900		3.61105
77 1,1,1,2-Tetrachloroethane	0.38736	0.38448	0.38813	0.40500	0.43920	0.42882					
	0.44083	0.41949	0.42308				AVRG		0.41293		5.42031
78 Ethylbenzene	0.46863	0.44438	0.43900	0.44868	0.46893	0.46974					
	0.48635	0.45370	0.46020				AVRG		0.45996		3.25757
79 p,m-Xylene	1.08360	1.02516	1.03021	1.02647	1.09248	1.10006					
	1.14091	1.05148	1.08004				AVRG		1.07005		3.70207
80 o-Xylene	1.03543	0.94634	0.93975	0.96149	1.02903	1.02348					
	1.06366	0.98746	0.99964				AVRG		0.99848		4.31239
81 Styrene	0.72784	0.69309	0.67601	0.70486	0.77158	0.76385					
	0.79388	0.73651	0.75509				AVRG		0.73586		5.31379
82 Bromoform	0.13141	0.13387	0.14083	0.15492	0.17588	0.17323					
	0.17993	0.17198	0.17501				AVRG		0.15967		12.29971
83 Isopropylbenzene	3.85331	3.74328	3.61636	3.65693	3.84165	3.82441					
	3.94888	3.82941	3.79850				AVRG		3.79030		2.71579

INITIAL CALIBRATION DATA

Start Cal Date : 03-SEP-2002 16:36
End Cal Date : 04-SEP-2002 05:13
Quant Method : ISTD
Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gc16.i/090402_25ml6w_ical2.b/25ml6w.m
Cal Date : 20-Sep-2002 11:40 beeson

Compound	0.5000 Level 1	1 Level 2	2 Level 3	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
	14 Level 7	20 Level 8	40 Level 9								
84 Cyclohexanone	+++++	0.01439	0.01223	0.01183	0.01423	0.00835					
	0.00906	0.01131	0.01240				AVRG		0.01172		18.45670 <-
86 1,1,2,2-Tetrachloroethane	0.43627	0.43994	0.41435	0.41608	0.44764	0.45138					
	0.46530	0.44661	0.44170				AVRG		0.43992		3.69894
87 Bromobenzene	0.83789	0.80309	0.78335	0.79138	0.85110	0.83909					
	0.85552	0.83495	0.81701				AVRG		0.82371		3.17448
88 trans-1,4-Dichloro-2-butene	+++++	0.04006	0.03397	0.04035	0.03915	0.03997					
	0.04040	0.04158	0.04223				AVRG		0.03971		6.32488
89 1,2,3-Trichloropropane	0.11264	0.10836	0.09902	0.10156	0.11283	0.10773					
	0.11045	0.10908	0.10663				AVRG		0.10759		4.34425
90 n-Propylbenzene	4.71647	4.43560	4.27986	4.32397	4.62684	4.61627					
	4.75229	4.49461	4.55052				AVRG		4.53294		3.62943
91 2-Chlorotoluene	2.79144	2.67380	2.59295	2.60773	2.75970	2.75567					
	2.82818	2.67041	2.70430				AVRG		2.70935		2.98199
92 1,3,5-Trimethylbenzene	2.74484	2.60556	2.54395	2.62663	2.79334	2.76719					
	2.86563	2.70901	2.73931				AVRG		2.71061		3.73369
93 4-Chlorotoluene	2.99357	2.74676	2.74577	2.71983	2.85795	2.88932					
	2.97619	2.78664	2.79864				AVRG		2.83496		3.55440

INITIAL CALIBRATION DATA

Start Cal Date : 03-SEP-2002 16:36
End Cal Date : 04-SEP-2002 05:13
Quant Method : ISTD
Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gc16.i/090402_25ml6w_ical2.b/25ml6w.m
Cal Date : 20-Sep-2002 11:40 beeson

Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
94 tert-Butylbenzene	3.35689	3.24019	3.08606	3.15558	3.32478	3.30799					
	3.41903	3.27619	3.28595				AVRG		3.27252		3.09850
95 Pentachloroethane	+++++	0.17963	0.19286	0.21705	0.21213	0.23532					
	0.22747	0.22370	0.22641				AVRG		0.21432		8.86035
96 1,2,4-Trimethylbenzene	2.54668	2.39968	2.36464	2.40152	2.54693	2.55818					
	2.63501	2.43782	2.45373				AVRG		2.48269		3.69682
97 sec-Butylbenzene	4.91570	4.59409	4.42433	4.53056	4.75875	4.77394					
	4.94519	4.69174	4.74716				AVRG		4.70905		3.62438
98 1,3-Dichlorobenzene	1.57285	1.51503	1.47793	1.48569	1.58959	1.58054					
	1.63299	1.56874	1.55730				AVRG		1.55341		3.27954
99 p-Isopropyltoluene	3.52675	3.30374	3.29214	3.33214	3.50086	3.54397					
	3.67306	3.48773	3.50119				AVRG		3.46240		3.68067
101 1,4-Dichlorobenzene	1.65683	1.54618	1.44381	1.43190	1.53119	1.52426					
	1.57175	1.48176	1.50972				AVRG		1.52193		4.48828
102 n-Butylbenzene	3.61517	2.95755	3.00693	3.03937	3.30688	3.36131					
	3.46513	3.19926	3.23011				AVRG		3.24241		6.78055
103 1,2-Dichlorobenzene	1.19871	1.10039	1.05455	1.08662	1.17411	1.16897					
	1.19659	1.13386	1.12951				AVRG		1.13814		4.44649

INITIAL CALIBRATION DATA

Start Cal Date : 03-SEP-2002 16:36
End Cal Date : 04-SEP-2002 05:13
Quant Method : ISTD
Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gc16.i/090402_25ml6w_ical2.b/25ml6w.m
Cal Date : 20-Sep-2002 11:40 beeson

Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
104 1 2 Dibromo-3-Chloropropane	3265	5189	8960	19793	37720	47332					
	63888	88583	173297				LINR	-0.03792	0.06187		0.99867
M 105 Xylene (total)	1.03543	0.94634	0.93975	0.96149	1.02903	1.02348					
	1.06366	0.98746	0.99964				AVRG		0.99848		4.31239
106 1,3,5-Trichlorobenzene	+++++	1.04319	1.08304	1.05171	1.22476	1.21747					
	1.21573	1.18781	1.19108				AVRG		1.15185		6.81249
107 1,2,4-Trichlorobenzene	0.73967	0.66124	0.66396	0.68413	0.72302	0.74928					
	0.74564	0.71705	0.71889				AVRG		0.71143		4.75110
108 Hexachlorobutadiene	1.04898	0.87466	0.90546	0.91523	0.96182	0.98593					
	1.00436	0.95111	0.94848				AVRG		0.95511		5.59204
109 Naphthalene	0.61701	0.42357	0.43206	0.44234	0.50338	0.52861					
	0.55214	0.48929	0.51793				AVRG		0.50070		12.52319
110 1,2,3-Trichlorobenzene	0.54795	0.48344	0.47340	0.48008	0.53329	0.53322					
	0.54786	0.51428	0.51555				AVRG		0.51434		5.65770
111 2-Methylnaphthalene	+++++	8562	12605	49429	113968	154109					
	215823	314686	584747				LINR	0.06389	0.22119		0.99671

INITIAL CALIBRATION DATA

Start Cal Date : 03-SEP-2002 16:36
End Cal Date : 04-SEP-2002 05:13
Quant Method : ISTD
Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gc16.i/090402_25ml6w_ical2.b/25ml6w.m
Cal Date : 20-Sep-2002 11:40 beeson

Compound	0.5000 Level 1 / 14 Level 7	1 Level 2 / 20 Level 8	2 Level 3 / 40 Level 9	5 Level 4	8 Level 5	10 Level 6	Curve	Coefficients b	m1	m2	%RSD or R^2
$ 38 Dibromofluoromethane	0.62426	0.62689	0.59883	0.58905	0.57449	0.58121					
	0.60429	0.59655	0.63566				AVRG		0.60347		3.52983
$ 46 1,2-Dichloroethane-d4	0.13821	0.14374	0.13568	0.12921	0.13110	0.13482					
	0.13518	0.13361	0.13877				AVRG		0.13559		3.18286
$ 63 Toluene-d8	0.79815	0.80377	0.79859	0.77898	0.78005	0.80514					
	0.82448	0.81143	0.85300				AVRG		0.80595		2.81106
$ 85 p-Bromofluorobenzene	0.66688	0.60303	0.58396	0.59553	0.58209	0.59800					
	0.60057	0.59267	0.61080				AVRG		0.60373		4.19427

INITIAL CALIBRATION DATA

Start Cal Date : 03-SEP-2002 16:36
End Cal Date : 04-SEP-2002 05:13
Quant Method : ISTD
Target Version : 3.50
Integrator : HP RTE
Method file : /var/chem/gc16.i/090402_25ml6w_ical2.b/25ml6w.m
Cal Date : 20-Sep-2002 11:40 beeson

Average %RSD Results.
Calculated Average %RSD = 4.79873943
Maximum Average %RSD = 15
* Passed Average %RSD Test.

Curve	Formula	Units
Averaged	Amt = Rsp/ml	Response
Linear	Amt = b + Rsp/ml	Response

CONTINUING CALIBRATION COMPOUNDS

Instrument ID: gc13.i
Lab File ID: 3c1017.d
Analysis Type: WATER
Lab Sample ID: VSTD010
Method: /var/chem/gc13.i/101802.b/hydr3w.m

Injection Date: 18-OCT-2002 08:19
Init. Cal. Date(s): 09-OCT-2002 18-OCT-2002
Init. Cal. Times: 01:03 04:12
Quant Type: ISTD

COMPOUND	RRF / AMOUNT	RF10	CCAL RRF10	MIN RRF	%D / %DRIFT	MAX %D / %DRIFT	CURVE TYPE
1 Dichlorodifluoromethane	0.56178	0.54856	0.54856	0.010	2.35379	25.00000	Averaged
2 Chloromethane	0.25846	0.24058	0.24058	0.100	6.91980	25.00000	Averaged
3 Vinyl chloride	0.30306	0.29342	0.29342	0.010	3.17939	20.00000	Averaged
4 Bromomethane	10.40777	10.00000	0.25389	0.010	-4.07775	25.00000	Linear
5 Chloroethane	0.19849	0.19521	0.19521	0.010	1.65178	25.00000	Averaged
6 Trichlorofluoromethane	0.70821	0.65375	0.65375	0.010	7.69061	25.00000	Averaged
7 Acrolein	0.00277	0.00267	0.00267	0.001	3.49417	50.00000	Averaged
8 1,1-Dichloroethene	0.34860	0.33105	0.33105	0.010	5.03428	20.00000	Averaged
9 Trichlorotrifluoroethane	0.62178	0.61637	0.61637	0.010	0.86890	25.00000	Averaged
10 Acetone	0.01768	0.01751	0.01751	0.010	0.97800	50.00000	Averaged
12 Carbon Disulfide	1.08796	0.97272	0.97272	0.010	10.59231	50.00000	Averaged
13 Acetonitrile	0.00403	0.00348	0.00348	0.010	13.64136	50.00000	Averaged <-
14 Methylene chloride	0.26819	0.25115	0.25115	0.010	6.35439	25.00000	Averaged
16 Acrylonitrile	0.01247	0.01217	0.01217	0.010	2.38988	50.00000	Averaged
17 trans-1,2-Dichloroethene	0.38662	0.38320	0.38320	0.010	0.88388	25.00000	Averaged
18 Methyl-tert-Butyl Ether	0.23934	0.22761	0.22761	0.010	4.90007	25.00000	Averaged
21 1,1-Dichloroethane	0.63032	0.60963	0.60963	0.100	3.28208	25.00000	Averaged
22 Vinyl Acetate	0.09111	0.07664	0.07664	0.010	15.87596	50.00000	Averaged
27 2,2-Dichloropropane	0.56107	0.55119	0.55119	0.010	1.76090	25.00000	Averaged
26 cis-1,2-Dichloroethene	0.32530	0.33070	0.33070	0.010	-1.66138	25.00000	Averaged
28 2-Butanone	0.02133	0.01666	0.01666	0.010	21.89598	50.00000	Averaged
29 Propionitrile	0.00353	0.00349	0.00349	0.010	1.34156	50.00000	Averaged <-
30 Bromochloromethane	0.13282	0.13717	0.13717	0.010	-3.27280	25.00000	Averaged
31 Tetrahydrofuran	0.01284	0.01229	0.01229	0.010	4.28371	25.00000	Averaged
32 Chloroform	0.67739	0.67669	0.67669	0.010	0.10367	20.00000	Averaged
$ 33 Dibromofluoromethane	0.50513	0.50328	0.50328	0.010	0.36720	25.00000	Averaged
35 1,1,1-Trichloroethane	0.67800	0.65160	0.65160	0.010	3.89408	25.00000	Averaged
37 1,1-Dichloropropene	0.61267	0.57582	0.57582	0.010	6.01531	25.00000	Averaged
38 Carbon tetrachloride	0.60852	0.56106	0.56106	0.010	7.79913	25.00000	Averaged
$ 39 1,2-Dichloroethane-d4	0.16231	0.15520	0.15520	0.010	4.38300	25.00000	Averaged
40 Benzene	0.92161	0.88617	0.88617	0.010	3.84500	25.00000	Averaged
41 1,2-Dichloroethane	0.17841	0.17635	0.17635	0.010	1.15337	25.00000	Averaged
46 Trichloroethene	0.46811	0.45876	0.45876	0.010	1.99793	25.00000	Averaged
49 1,2-Dichloropropane	0.26329	0.26861	0.26861	0.010	-2.02030	20.00000	Averaged
51 Dibromomethane	0.15087	0.15544	0.15544	0.010	-3.03131	25.00000	Averaged
52 Bromodichloromethane	0.48062	0.49505	0.49505	0.010	-3.00110	25.00000	Averaged

CONTINUING CALIBRATION COMPOUNDS

Instrument ID: gc13.i
Lab File ID: 3c1017.d
Analysis Type: WATER
Lab Sample ID: VSTD010
Method: /var/chem/gc13.i/101802.b/hydr3w.m

Injection Date: 18-OCT-2002 08:19
Init. Cal. Date(s): 09-OCT-2002 18-OCT-2002
Init. Cal. Times: 01:03 04:12
Quant Type: ISTD

COMPOUND	RRF / AMOUNT	RF10	CCAL RRF10	MIN RRF	%D / %DRIFT	MAX %D / %DRIFT	CURVE TYPE
56 2-Chloroethylvinylether	0.04719	0.04546	0.04546	0.010	3.66493	25.00000	Averaged
57 cis-1.3-Dichloropropene	0.35118	0.36223	0.36223	0.010	-3.14488	25.00000	Averaged
58 4-Methyl-2-pentanone	0.05638	0.04753	0.04753	0.010	15.68973	50.00000	Averaged
$ 60 Toluene-d8	0.88400	0.89680	0.89680	0.010	-1.44730	25.00000	Averaged
62 Toluene	9.88100	10.00000	0.60931	0.010	1.18997	20.00000	Linear
63 trans-1.3-Dichloropropene	0.22272	0.23250	0.23250	0.010	-4.39171	25.00000	Averaged
64 1.1.2-Trichloroethane	0.12911	0.13763	0.13763	0.010	-6.60241	25.00000	Averaged
65 1.3-Dichloropropane	0.32475	0.34115	0.34115	0.010	-5.05224	25.00000	Averaged
66 Tetrachloroethene	0.79002	0.78952	0.78952	0.010	0.06293	25.00000	Averaged
67 2-Hexanone	0.04918	0.03736	0.03736	0.010	24.02263	50.00000	Averaged
69 Dibromochloromethane	0.41518	0.44149	0.44149	0.010	-6.33647	25.00000	Averaged
70 1.2-Dibromoethane	0.17613	0.18706	0.18706	0.010	-6.20662	25.00000	Averaged
71 1-Chlorohexane	0.52830	0.52226	0.52226	0.010	1.14384	25.00000	Averaged
72 Chlorobenzene	0.98323	1.01837	1.01837	0.300	-3.57418	25.00000	Averaged
73 1.1.1.2-Tetrachloroethane	0.48194	0.50779	0.50779	0.010	-5.36268	25.00000	Averaged
74 Ethylbenzene	0.52025	0.53375	0.53375	0.010	-2.59605	20.00000	Averaged
75 p.m-Xylene	1.43762	1.46823	1.46823	0.010	-2.12930	25.00000	Averaged
M 77 1.2-Dichloroethene (total)	0.35596	0.35695	0.35695	0.010	-0.27914	25.00000	Averaged
78 o-Xylene	1.31381	1.35062	1.35062	0.010	-2.80176	25.00000	Averaged
79 Styrene	0.84104	0.91004	0.91004	0.010	-8.20461	25.00000	Averaged
80 Bromoform	0.20355	0.22597	0.22597	0.100	-11.01394	25.00000	Averaged
81 Isopropylbenzene	3.38045	3.38829	3.38829	0.010	-0.23205	25.00000	Averaged
$ 82 p-Bromofluorobenzene	0.70610	0.72322	0.72322	0.010	-2.42485	25.00000	Averaged
83 1.1.2.2-Tetrachloroethane	0.41264	0.43197	0.43197	0.300	-4.68397	25.00000	Averaged
84 Bromobenzene	0.81645	0.85720	0.85720	0.010	-4.99194	25.00000	Averaged
85 1.2.3-Trichloropropane	0.08772	0.09149	0.09149	0.010	-4.29380	25.00000	Averaged
86 n-Propylbenzene	4.26490	4.20759	4.20759	0.010	1.34364	25.00000	Averaged
87 2-Chlorotoluene	3.01077	2.98657	2.98657	0.010	0.80387	25.00000	Averaged
88 1.3.5-Trimethylbenzene	2.54751	2.61229	2.61229	0.010	-2.54317	25.00000	Averaged
89 4-Chlorotoluene	2.94200	3.02366	3.02366	0.010	-2.77565	25.00000	Averaged
92 tert-Butylbenzene	2.95820	2.98126	2.98126	0.010	-0.77960	25.00000	Averaged
93 1.2.4-Trimethylbenzene	2.40871	2.46613	2.46613	0.010	-2.38388	25.00000	Averaged
94 sec-Butylbenzene	4.15921	4.17269	4.17269	0.010	-0.32413	25.00000	Averaged
95 1.3-Dichlorobenzene	1.55533	1.57882	1.57882	0.010	-1.51011	25.00000	Averaged
96 p-Isopropyltoluene	3.22249	3.26814	3.26814	0.010	-1.41647	25.00000	Averaged

CONTINUING CALIBRATION COMPOUNDS

Instrument ID: gcl3.i
Lab File ID: 3c1017.d
Analysis Type: WATER
Lab Sample ID: VSTD010
Method: /var/chem/gcl3.i/101802.b/hydr3w.m

Injection Date: 18-OCT-2002 08:19
Init. Cal. Date(s): 09-OCT-2002 18-OCT-2002
Init. Cal. Times: 01:03 04:12
Quant Type: ISTD

COMPOUND	RRF / AMOUNT	RF10	CCAL RRF10	MIN RRF	%D / %DRIFT	MAX %D / %DRIFT	CURVE TYPE
98 1,4-Dichlorobenzene	1.71550	1.77446	1.77446	0.010	-3.43665	25.00000	Averaged
99 n-Butylbenzene	3.38841	3.41990	3.41990	0.010	-0.92945	25.00000	Averaged
101 1,2-Dichlorobenzene	1.19886	1.24386	1.24386	0.010	-3.75421	25.00000	Averaged
102 1,2-Dibromo-3-Chloropropane	0.06900	0.07251	0.07251	0.010	-5.09657	25.00000	Averaged
104 1,2,4-Trichlorobenzene	0.78450	0.80761	0.80761	0.010	-2.94610	25.00000	Averaged
105 Hexachlorobutadiene	1.04401	1.05231	1.05231	0.010	-0.79476	25.00000	Averaged
106 Naphthalene	0.46186	0.48093	0.48093	0.010	-4.12837	25.00000	Averaged
107 1,2,3-Trichlorobenzene	0.55649	0.57018	0.57018	0.010	-2.46004	25.00000	Averaged
M 108 Xylene (total)	1.31381	1.35062	1.35062	0.010	-2.80176	25.00000	Averaged

Average %D / Drift Results	
Calculated Average %D/Drift =	4.22211
Maximum Average %D/Drift =	15.00000
* Passed Average %D/Drift Test.	

VOLATILE REPORT SW846 METHOD 8260B WATERS

Data file : /var/chem/gcl3.i/101802.b/3c1017.d
Lab Smp Id: VSTD010 Client Smp ID: VSTD010
Inj Date : 18-OCT-2002 08:19
Operator : DCT Inst ID: gcl3.i
Smp Info : CV V02J18DAA
Misc Info : VSTD010,1,7
Comment : HP 5971, 5890 GC TEKMAR 3000/2016
Method : /var/chem/gcl3.i/101802.b/hydr3w.m
Meth Date : 21-Oct-2002 14:16 petruszj Quant Type: ISTD
Cal Date : 18-OCT-2002 02:22 Cal File: 3i1017i.d
Als bottle: 7 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: ICAL1.sub
Target Version: 3.50
Processing Host: manatee

Concentration Formula: Amt * DF * Uf * 1/Vo * CpndVariable

Name	Value	Description
DF	1.00000	Dilution Factor
Uf	25.00000	ng unit correction factor
Vo	25.00000	Sample Volume purged (mL)
Cpnd Variable		Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ug/L)	AMOUNTS ON-COL (ug/L)
1 Dichlorodifluoromethane	85	2.010	2.010	(0.308)	1061832	10.0000	9.765
2 Chloromethane	50	2.228	2.228	(0.342)	465677	10.0000	9.308
3 Vinyl chloride	62	2.337	2.337	(0.358)	567967	10.0000	9.682
4 Bromomethane	94	2.709	2.709	(0.415)	491442	10.0000	10.408
5 Chloroethane	64	2.827	2.827	(0.434)	377863	10.0000	9.835
6 Trichlorofluoromethane	101	3.144	3.144	(0.482)	1265436	10.0000	9.231
7 Acrolein	56	3.571	3.571	(0.548)	413405	800.000	772.05
8 1,1-Dichloroethene	96	3.716	3.716	(0.570)	640800	10.0000	9.496
9 Trichlorotrifluoroethane	101	3.725	3.725	(0.571)	1193097	10.0000	9.913
10 Acetone	43	3.734	3.734	(0.573)	33885	10.0000	9.902
12 Carbon Disulfide	76	3.970	3.970	(0.609)	1882867	10.0000	8.941
13 Acetonitrile	41	4.052	4.052	(0.621)	107700	160.000	138.17
14 Methylene chloride	84	4.252	4.252	(0.652)	486135	10.0000	9.364
16 Acrylonitrile	53	4.515	4.515	(0.692)	376900	160.000	156.18
17 trans-1,2-Dichloroethene	96	4.588	4.588	(0.704)	741748	10.0000	9.912

						AMOUNTS	
Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ug/L)	ON-COL (ug/L)
18 Methyl-tert-Butyl Ether	73	4.588	4.588	(0.704)	440581	10.0000	9.510
21 1,1-Dichloroethane	63	5.087	5.087	(0.780)	1180051	10.0000	9.672
22 Vinyl Acetate	43	5.159	5.159	(0.791)	148354	10.0000	8.412
27 2,2-Dichloropropane	77	5.822	5.822	(0.893)	1066914	10.0000	9.824
26 cis-1,2-Dichloroethene	96	5.813	5.813	(0.891)	640135	10.0000	10.166
28 2-Butanone	43	5.822	5.822	(0.893)	32250	10.0000	7.810(M)
29 Propionitrile	54	5.885	5.885	(0.788)	115091	160.000	157.85
30 Bromochloromethane	128	6.121	6.121	(0.939)	265518	10.0000	10.327
31 Tetrahydrofuran	42	6.176	6.176	(0.947)	237949	100.000	95.716
32 Chloroform	83	6.221	6.221	(0.954)	1309841	10.0000	9.990
$ 33 Dibromofluoromethane	113	6.430	6.430	(0.986)	974179	10.0000	9.963
35 1,1,1-Trichloroethane	97	6.475	6.475	(0.993)	1261279	10.0000	9.610
37 1,1-Dichloropropene	75	6.693	6.693	(1.026)	1114596	10.0000	9.398
38 Carbon tetrachloride	117	6.702	6.702	(0.898)	1157793	10.0000	9.220
$ 39 1,2-Dichloroethane-d4	65	6.866	6.866	(0.920)	320267	10.0000	9.562
40 Benzene	78	6.965	6.965	(0.933)	1828685	10.0000	9.616
41 1,2-Dichloroethane	62	6.956	6.956	(0.932)	363917	10.0000	9.885
* 42 Pentafluorobenzene	168	6.521	6.521	(1.000)	1935670	10.0000	
46 Trichloroethene	130	7.809	7.809	(1.046)	946684	10.0000	9.800
* 48 1,4-Difluorobenzene	114	7.465	7.465	(1.000)	2063579	10.0000	
49 1,2-Dichloropropane	63	8.091	8.091	(1.084)	554292	10.0000	10.202
51 Dibromomethane	93	8.236	8.236	(1.103)	320764	10.0000	10.303
52 Bromodichloromethane	83	8.445	8.445	(1.131)	1021568	10.0000	10.300
56 2-Chloroethylvinylether	63	8.871	8.871	(1.188)	93818	10.0000	9.634
57 cis-1,3-Dichloropropene	75	9.053	9.053	(1.213)	747482	10.0000	10.314
58 4-Methyl-2-pentanone	43	9.261	9.261	(1.241)	98083	10.0000	8.431
$ 60 Toluene-d8	98	9.425	9.425	(1.263)	1850610	10.0000	10.145
62 Toluene	92	9.516	9.516	(1.275)	1257356	10.0000	9.881
63 trans-1,3-Dichloropropene	75	9.806	9.806	(1.314)	479783	10.0000	10.439
64 1,1,2-Trichloroethane	97	10.051	10.051	(1.541)	266408	10.0000	10.660
65 1,3-Dichloropropane	76	10.278	10.278	(0.901)	457036	10.0000	10.505
66 Tetrachloroethene	166	10.278	10.278	(0.901)	1057706	10.0000	9.994
67 2-Hexanone	43	10.414	10.414	(0.913)	50056	10.0000	7.598
69 Dibromochloromethane	129	10.596	10.596	(0.928)	591453	10.0000	10.634
70 1,2-Dibromoethane	107	10.750	10.750	(1.440)	386007	10.0000	10.621
71 1-Chlorohexane	91	11.431	11.431	(1.753)	1010915	10.0000	9.886
72 Chlorobenzene	112	11.449	11.449	(1.003)	1364294	10.0000	10.357
73 1,1,1,2-Tetrachloroethane	131	11.567	11.567	(1.014)	680273	10.0000	10.536
74 Ethylbenzene	106	11.621	11.621	(1.018)	715059	10.0000	10.260
75 p,m-Xylene	91	11.784	11.784	(1.033)	3933906	20.0000	20.426
* 76 Chlorobenzene-d5	117	11.412	11.412	(1.000)	1339678	10.0000	
M 77 1,2-Dichloroethene (total)	96				1381883	20.0000	20.056
78 o-Xylene	91	12.347	12.347	(1.082)	1809390	10.0000	10.280
79 Styrene	104	12.365	12.365	(1.083)	1219166	10.0000	10.820
80 Bromoform	173	12.610	12.610	(1.105)	302731	10.0000	11.101
81 Isopropylbenzene	105	12.883	12.883	(0.872)	2562052	10.0000	10.023
$ 82 p-Bromofluorobenzene	95	13.091	13.091	(1.147)	968882	10.0000	10.242

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ug/L)	ON-COL (ug/L)
83 1,1,2,2-Tetrachloroethane	83	13.291	13.291	(0.900)	326632	10.0000	10.468
84 Bromobenzene	156	13.300	13.300	(0.900)	648172	10.0000	10.499
85 1,2,3-Trichloropropane	110	13.346	13.346	(0.904)	69180	10.0000	10.429(M)
86 n-Propylbenzene	91	13.473	13.473	(0.912)	3181561	10.0000	9.866
87 2-Chlorotoluene	91	13.581	13.581	(0.920)	2258288	10.0000	9.920
88 1,3,5-Trimethylbenzene	105	13.736	13.736	(0.930)	1975281	10.0000	10.254
89 4-Chlorotoluene	91	13.745	13.745	(0.931)	2286335	10.0000	10.278
92 tert-Butylbenzene	119	14.208	14.208	(0.962)	2254274	10.0000	10.078
93 1,2,4-Trimethylbenzene	105	14.280	14.280	(0.967)	1864759	10.0000	10.238
94 sec-Butylbenzene	105	14.534	14.534	(0.984)	3155174	10.0000	10.032
95 1,3-Dichlorobenzene	146	14.680	14.680	(0.994)	1193822	10.0000	10.151
96 p-Isopropyltoluene	119	14.752	14.752	(0.999)	2471195	10.0000	10.142
98 1,4-Dichlorobenzene	146	14.807	14.807	(1.002)	1341752	10.0000	10.344
99 n-Butylbenzene	91	15.351	15.351	(1.039)	2585952	10.0000	10.093
* 100 1,4-Dichlorobenzene-d4	152	14.770	14.770	(1.000)	756148	10.0000	
101 1,2-Dichlorobenzene	146	15.351	15.351	(1.039)	940546	10.0000	10.375
102 1,2-Dibromo-3-Chloropropane	75	16.449	16.449	(1.114)	54832	10.0000	10.510
104 1,2,4-Trichlorobenzene	180	17.502	17.502	(1.185)	510672	10.0000	10.295
105 Hexachlorobutadiene	225	17.720	17.720	(1.200)	795702	10.0000	10.079
106 Naphthalene	128	17.784	17.784	(1.204)	363651	10.0000	10.413
107 1,2,3-Trichlorobenzene	180	18.056	18.056	(1.222)	431137	10.0000	10.246
M 108 Xylene (total)	91				5743296	10.0000	32.631

QC Flag Legend

M - Compound response manually integrated.

Surrogate Spike Recovery Default Limits

Surrogate Compound	Water	Low Soil
Toluene-d_8	88-110	81-117
4-Bromofluorobenzene	86-115	74-121
1,2-Dichloroethane-d_4	80-120	80-120
Dibromofluoromethane	86-118	80-120

Note: Above are Surrogate Spike Recovery Default Limits from 8260B. These limits are for guidance only, not intended as set limits. In-house generated statistical limits or QAPP limits will be used whenever possible. See Table 1 for in-house generated statistical limits.

LCS / MS Recovery Default Limits

MS Compound	Water	Soil/Sediment
1,1-Dichloroethene	61-145	59-172
Trichloroethene	71-120	62-137
Chlorobenzene	75-130	60-133
Toluene	76-125	59-139
Benzene	76-127	66-142

Internal standard areas must be within -50% to 100% of the EICP for the corresponding continuing calibration standards. Internal standard retention times must not deviate by 30%.

Attachment 3.

Example: GC/MS Volatiles/CAR Logbook; Tune Form; Sample Tracking Sheet, Maintenance Logbook

STL Chicago
GC/MS Volatile Analysis Logbook

Instrument ID# 16 CHI-22-20-049/A-05/02

Analysis Date / Time	File Name	Sample Number	Sample ID	Sparge No.	Sample Wt. / Vol.	Instr. Dil.	Int. Std. No.	pH < 2	Comments (MUST include SRN's)	Analyst Initials

Analyst Signature/Date:______________________ Reviewer Signature/Date:______________________ Page No.

Instrument ID# 16

STL Chicago
Corrective Action/Qualification Report GC/MS VOA

Analytical Methods **Tune Name:**__________'02

___SW846 8260/A/B ___EPA 524.2 __Other__________
___40CFR 624 ___OLM03.2
___OLM04.2 ___OLC02.1

ISTD/RT report
Initial calibration / Continuing Calibration IS#__________
(For method 524 include all points in ICAL)

Data File Name:__________

	IS 1	RT1	IS2	RT2	IS3	RT3	IS4	RT4
1								
2								
3								
4								

Tune Criteria
Description of Situation:__________
Action Taken:__________
Demonstration of Control:__________

Initial Calibration Cirteria
Description of Situation:__________
Action Taken:__________
Demonstration of Control__________

Continuing Calibration Criteria
Description of Situation:__________
Action Taken:__________
Demonstration of Control:__________

Internal Standards (continuing cal to continuing cal)
Description of situation:__________
Action Taken:__________
Demonstration of Control:__________

Method Blank
Description of situation:__________
Action Taken:__________
Demonstration of Control:__________

LCS
Description of Situation:__________
Action Taken:__________
Demonstration of Control:__________

Qualification of Data
Data Affected (Client/Sample #)__________
Qualification:__________

Associated samples reanalyzed: Yes No (see below)
Explanation for no reanalysis/data MUST be qualified and narrated:__________

Analyst Signature/date__________/__________

Reviewer Signature/date__________/__________

Tune Name: ________ STD CC: ________ Labnet ID: ________ Method: ________ Tune Batch: ________

Tune Time: ________ CC: ________ Labnet ID: ________ Method: ________ Tune Batch: ________

INST: ________ LCS: ________

Sample ID:	File:	Act	HT	pH	IS	SS	Misc. Info	Prep Method	Prep Batch	Analytical Batch

Exception Reports ☐

rpjah

Job Analysis History

10/21/2002

V2

Surrogate Reagent...:

Method Code....: 8260B Volatile Organics
Job Number.....: Customer Job ID..:
Project Number.:

Holding Time.....: 14 Day Holding Time
Customer.........:
Proj. Cat..: INDUSTRIAL PM...:

Job Report Type...: l2qfmdl
Contact.:
Hardcopy Due Date.: 10/21/2002 Fax Due Date.: 10/11/2002

Sample #	QC	Client Sample ID	Matrix	HT Date	TAT Date	File Name	Dil	Tune name	Action	Analst	Prep Batch	Comments
1	N	A90210BH01-01	SOIL	10/21/2002	10/21/2002							
2	N	A90210BH01-02	SOIL	10/21/2002	10/21/2002							
3	N	A90210BH01-GW	WATER	10/21/2002	10/21/2002							
13	N	A90210TB1	WATER	10/21/2002	10/21/2002							

Date of Maintenance:__________ Entry No.:__________

Analyst:__________

Description:__________

Follow-Up:__________

Analyst:__________ Date:__________

Date of Maintenance:__________ Entry No.:__________

Analyst:__________

Description:__________

Follow-Up:__________

Analyst:__________ Date:__________

Date of Maintenance:__________ Entry No.:__________

Analyst:__________

Description:__________

Follow-Up:__________

Analyst:__________ Date:__________

Reviewer Signature:__________ Date:__________

Attachment 4.

Evaluation and Acceptance Criteria Table

GCMS Calibration – Evaluation and Acceptance Criteria

Prepare standards	• The concentrations must cover the range of quantitation for all analytes • The lowest point of the calibration curve must be at, or below, the reporting limit	
Run calibration standards	• All analytes must contain at a minimum 5 calibration points for Linear regressions, linear curves, or average response factors • Analytes using second order fits must have a minimum of 6 calibration points on a curve • Analytes using 3rd order curves must have a minimum of 7 calibration points	• Calibration levels deemed to be statistical or visual outliers shall be dropped from the curve and replaced by a similar concentration standard. The standard shall be dropped in its entirety, not on a per analyte basis
Calculate "grand mean RSD" of all compounds	• The "grand mean RSD" is calculated before attempting to fit compounds to any calibration curves only because it is easier to do at this time. The "grand mean RSD" acceptance is only to be used as a last resort in determining whether a compound's calibration is acceptable. • Calibrations for compounds which occur on different dates (i.e. appix compounds analyzed at different dates than the HSL compounds), the "grand mean RSD" is calculated separately for each curve.	• If no %RSD for any given compound is above 15%, the "grand mean RSD will be less than 15%.
Evaluation each compound for linear fit, second order fit or 3rd order fit	• Having determined that the calibration meets minimum requirements, the laboratory will evaluate each compound to determine the best calibration fit using statistical and visual evaluation of the curve. • Using "priori" knowledge some compounds ranges may be shortened (i.e. not as low a reporting limit or not as high a range)	• Citing priori knowledge the laboratory may decide to "keep" calibrations for several compounds despite exceeding the statistical threshold allowed by the method. The compounds that fall in this category are known poor performers as indicated by the method or as indicated from historical performance data (e.g. Appendix IX compounds). These compounds will be listed in the OP as possible trouble analytes. Quantitation for these compounds may be biased and should be used only with caution.
Determine the best calibration approach for each compound	• RRF • Linear • 2nd order • Grand Mean RSD	• Even when the "grand mean RSD" indicates minimum acceptance has been met, the analyst must use discretion for quantification because some compounds may be biased. This approach must be used with caution.

Attachment 5.

Example: Analysis and Sample Tracking Flowcharts

ANALYSIS SCHEME FLOWCHART

(Terms defined in the Section 9)

```
                  PFTBA
                    |
                    |
                    |
                   BFB  (Form 5)
                  /  \ Every 12 hours
                 /    \
                /      \
Initial Calibration--------Continuing Calibration
   (Form 6)                 (Form 7)
                         Every 12 hours
                              |
                              |
                             \|/
                           Analysis
                              |
                              |
                             \|/
                Computer Data Production/Reduction
                              |
                              |
                             \|/
                 Operator Data Review/Reduction
                     (Form 1,2,3,4,5)
                              |
                              |
                             \|/
                  Final Review/Data Submission
                              |
                              |
                             \|/
                       Report Generation
```

Sample Tracking Flowchart (for EACH unique Job)

```
                    Sample Receipt
                         | \
                         |  \
                 Log-In    VOA Refrigerator
                    /   |        |
                   /    |        |
                  /     |        |
               COC   Computer|
                 |      |        |
                 |      |        |
                 |      |        |
                 |    Backlog    |
                 |      |        |
                 |\     |       /
                 | \    |      /
                 |  \   |     /
                 |   \  |    /
               VOAs    VOAs
                 |      |
                 |      |
               Filed   Posted
                  \     /
                   \   /
                    \/
                   Input
                     |
               Sample Tracking Board
                     |
                     |
                  Analysis
                     |
                     |
                  OUTPUT:   COC, Tune Form, Oper. Anal. Logbook, Comp.
                     | Data Prod., Tune, Calibration, QC
               ______|______
· Operator Review/Data Reduction        · Archival (automated scheduled back-ups)
· Data Package w/ Review Form, CARs     · Tune Form filed
· 1st 100% Review                       · Review Form, CAR copies filed
· Submission/Final Review               · Data purged off system
        |_______________________________________|
                         |
                 REPORT GENERATION
```

Attachment 6.

Example: Data Review Form

STL Chicago
GCMS DATA REVIEW CHECKLIST

Site Name:______________________ Primary Reviewer:______________________ Review Date:______________
JOB Number ______________________ Secondary Reviewer:______________________ Review Date:______________
No. of Samples/Matrix: a) ______ WATER b) ______ SOIL c) ______ SPLP / TCLP d) ______ Other (________________)
Method: a) VOA ____________ 5030______ Encores: 5035–High ______ 5035-Low______ b) BNA ______________
Report Type: a) MDL U b) RL U c) Breach d) P1 / P2 ("QCORG" printed QC must match PM selected Report Type)

TASK	PRI REV	SEC REV	COMMENTS
LAB CHRON: 1) Matches Big Board (Job Analysis History)			
2) Matches Raw Data (Form 4 / 5)			
3) Note Sample dilutions and list reason. a) High Sample Conc. b) Interference present IF original and re-run are to be reported in LabNet Re-log Samples (Indicate data type used) Re-Analyzed (RE) Re-Extracted (RA) Dilution (DL)			Smp # Original Dilution Comments BNA Only: Final Volume Adjustment _________
4) Sample Hold Times Met (SDR written: Yes No)			
5) Proper Prep Links Created S-F6: Routine Preps; 5035PL; 5035PH S-F9: TCLP; SPLP ; 5035 Archon Purge & Trap			
Incomplete JOB Status Report reveals No Outstanding Data			
PROJ. REQ. MET: 1) Sample Detection Limit Met			
2) Reported J values meet reporting criteria			
3) Method Blank Detection Limits Met (MB<1/2 RL for LCG)			
4) Project Specific Compounds of Concern : Yes No			
LabNet Report matches Quant Report: Sample weights / Volumes / % Moisture / Factors verified			
Manual Integration documentation / verification complete			
FORM 2: Surrogate Recoveries Within Limits Statistical Limits ____ Method Limits ____ Project Limits ____ (S-F10 used to Clone By Project) AFCEE___; LCG___; QAPP___ Directed Note to PM: Yes / No			Smp # Original Re-analysis Comments
FORM 3: MS/MSD Recoveries Acceptable Statistical Limits ____ Method Limits ____ Project Limits ____ (S-F10 used to Clone By Project) AFCEE___; LCG___; QAPP___ Directed Note to PM: Yes / No			Smp # ______ MS MSD
FORM 3: LCS Recoveries Acceptable (LCD if no MS/MSD) Statistical Limits ____ Method Limits ____ Project Limits ____ (S-F10 used to Clone By Project) AFCEE___; LCG___; QAPP___ Directed Note to PM: Yes / No			Batch # ________ Batch # ________ Batch # ________ Batch # ________ Batch # ________ Batch # ________

TASK	PRI REV	SEC REV	COMMENTS
FORM 5: Tuning Criteria Met			
FORM 6: Initial Calibration Criteria Met			
ICAL Spike Required: Yes No Control Limit applied: ________________			
FORM 7: Daily Calibration (CCV) Criteria Met			
MRL Check Required: Yes No (LCG Requirement – Before and after Sample analysis) Control Limit Applied: ________________			Before: After: Batch # _________ Batch # _________ Batch # _________ Batch # _________ Batch # _________
FORM 8: Internal Standards Criteria Met Default – Internal Standards checked against the continuing calibration LCC - Internal Standards checked against the mid-point of the ICAL *__Form 8 MUST BE verified for correct areas present: YES NO__* **It is critical that the ICAL1 be processed last. The last ICAL processed is held in the method which is what appears on the target IS report.** Directed Note to PM: Yes / No			Smp # Original Re-analysis Comments
Lat Net Batch Status Report Displays Data at RPT / RVWD Status	RPT	RVWD	
RAW DATA: 1) Raw Data Verified/Complete			
2) Raw Data Matches Forms			
3) 5035 Prep Log page present / verified			
4) Quant Report Matches Spectra			
5) Manual integration reports (befores and afters) present (when required by client) and reason correctly documented and approved. Manual Integration Summary Printed: Yes No			File ID:
NARRATIVE: 1) Holding Times			
2) Method References			
3) % Recoveries / RPD's			
4) Analytical Difficulties/Typos/CAR's Directed Note to PM: Yes / No			
Manual Calculation of On Column result: Response Factor (Smp) x Concentration of IS IS Response Factor (Smp) Cmpd. RRF (Cont.Calib)			Sample : ___________ Compound: ________________

Additional Comments:

SOP No.	Revision No.	Date	Page
UME-6010B	04	10/25/02	1 of 25

TITLE: Metals Analysis
Trace Inductively Coupled Argon Plasma by SW-846 6010B (Simultaneous Operation)

Updated by:	Signature:	Date:
Paul F. Kolarczyk Senior Analyst	Paul F. Kolarczyk	10/17/2002

Approved by:	Signature:	Date:
Mani S. Iyer Section Manager, Metals Dept.	Mani S. Iyer	10/17/02
David L. Kaczka Env. Health & Safety Coor.	DLKaczka	10-21-02
Terese A. Preston Quality Manager	Terese A. Preston	10-21-02

CONTROLLED DISTRIBUTION

COPY # : Uncontrolled

ISSUED TO : Mark Densmore, Secor
Re: UTC Proposal

Full Signature Approvals Are Kept on File
with STL's QA Standard Practice Records

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for determining metal concentrations by Trace Inductively Coupled Argon Plasma (ICAP) Emission Spectrometry - Simultaneous Operation. This SOP was written using U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste", Third Edition, Method 6010B as a reference.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

1.1.2 Instrument Detection Limits

Instrument Detection Limits (IDLs) are performed on a quarterly basis for each element and for each instrument (as specified in CLP). These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined. (Note: The annual MDL may be used in lieu of one of the semi-annual IDL sets, providing required reporting limits are achieved).

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values ~3-5x the respective MDL to ensure confidence in the value reported.

Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement. Refer to Table 1 for element wavelength and reporting limits.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM).

1.2 Summary of Method

ICAP is a technique for the analysis of soluble or digested samples for metal concentrations using atomic emission spectrometry. All matrices, including water, TCLP extracts, wastes, soils, sludges and sediments, require digestion prior to analysis. The instrument is capable of analyzing simultaneously 31 different elements on a sample.

2.0 INTERFERENCES

Spectral, Physical and Chemical Interferences are the three main interferences that are commonly present on the ICAP.

2.1 Spectral Interferences

Mainly caused by continuous background wavelength, stray light from a high concentration element or overlap of a spectral line from another element. The ICAP can correct for the first two types of interferences by using background correction adjacent to the wavelength. Spectral overlap can be corrected by monitoring the interfering wavelength and computer correcting the results for the false concentration. The values used to correct are known as Inter-element Correction Factors or IEC's.

2.2 Physical Interferences

Usually associated with the sample uptake and nebulization processes. These interferences can usually be eliminated by using a peristaltic pump which assures a constant sample uptake rate. If a sample is extremely viscous or contains a very high dissolved solids concentration, a dilution of the sample may be required to assure a constant and smooth nebulization rate.

2.3 Chemical Interferences

Normally not significant on the ICAP. These interferences include ionization effects and molecular compound formation. Chemical interferences are highly dependent on the sample matrix type and the element.

Trace ICP can have some ionization effects caused by torch positioning. To eliminate these effects, Cesium is added to the internal standard solution (100 mLs / 1 Liter).

Most interferences can be corrected by ensuring a constant sample uptake rate and by using the correcting abilities of the computer. If severe interferences are suspected, an alternate method such as Graphite Furnace Atomic Absorption (GFAA) can be used or to verify the ICAP results.

3.0 SAFETY

- Employees will adhere to the practices and policies in the STL Corporate Safety Manual (CSM) and will read the MSDSs for the materials used in this method before handling or using the material.
- If contact occurs with a standard containing Hydrofluoric Acid, flush with water and apply Calcium Gluconate Gel (located in standards cabinet) immediately. ***Seek medical attention.***
- The ICP torch puts out harmful ultraviolet radiation. The torch should never be looked at directly without proper eye protection (i.e. lens tinted for the wavelength of the torch.)
- Parts of the instrument can be extremely hot. Care should be taken if the instrument needs to be adjusted internally.
- People with pacemakers should not be near the ICP due to the radio frequency generator.
- Proper ventilation is required due to sample fumes and extreme heat generation (RF generator and plasma) and plasma emissions. People with medical conditions that may respond to ozone emissions should exercise caution.

4.0 EQUIPMENT AND SUPPLIES

4.1 Instrumentation

3 - Thermo Jarrell Ash ICAP 61E Trace Analyzer. These instruments are simultaneous ICAP's which currently have 31 analytical wavelengths. Additional wavelengths may be added as required.

The instruments are operated via desktop computers and Thermo Jarrell Ash software (Version 6.2). They also come equipped with a peristaltic pump for sample uptake and an autosampler.

4.2 Supplies

- Volumetric Flasks (Class A): 100 mLs; 1000 mLs
- Eppendorf Pipettes, varying volumes

5.0 REAGENTS AND STANDARDS

5.1 Reagents

- Milli-Q Water
- *Concentrated Nitric Acid (HNO_3) - InstraPure
- *Concentrated Hydrochloric Acid (HCl) - InstraPure

*Purchased from a vendor.

5.2 Standards and QC Solutions

All stock standards and QC solutions are purchased from an outside supplier in aqueous form. Two types of standards are used: single element and custom mixed standards. Single element standards are available for most elements at a 1,000 mg/L concentration. The shelf life of all purchased solutions are as stated by the manufacturer and are listed in LabNet (LIMS).

5.2.1 Calibration Standards

Prepared with Milli-Q water that has been acidified with 1% HNO_3 and 5% HCl. The calibration standards are prepared daily as follows:

1. **Calibration Blank**

Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. Repipette 10 mLs conc. HNO_3 and 50 mLs conc. HCl into the flask. Dilute to volume with Milli-Q water and mix thoroughly.

2. **Calibration Standards** (Refer to Appendix A for element concentrations)

S1: Add ~50 mLs of Milli-Q water to a 100 mL Class A volumetric flask. Repipette 1 mL conc. HNO_3 and 5 mLs conc. HCl into the flask. Using Eppendorf pipettes, add 1.0 mL each of RFW-ICPT-STD-1B, RFW-ICPT-STD-1C, and RFW-ICPT-STD-1D. Dilute to volume with Milli-Q water and mix thoroughly.

S1A: Add ~50 mLs of Milli-Q water to a 100 mL Class A volumetric flask. Repipette 1 mL conc. HNO_3 and 5 mLs conc. HCl into the flask. Using Eppendorf pipettes, add 0.4 mLs each RFW-ICPT-STD-1B, RFW-ICPT-STD-1C, and RFW-ICPT-STD-1D. Dilute to volume with Milli-Q water and mix thoroughly.

S1B: Add ~50 mLs of Milli-Q water to a 100 mL Class A volumetric flask. Repipette 1 mL conc. HNO_3 and 5 mLs conc. HCl into the flask. Using Eppendorf pipettes, add 0.5 mLs each of RFW-ICPT-STD-1B, RFW-ICPT-STD-1C, and RFW-ICPT-STD-1D. Dilute to volume with Milli-Q water and mix thoroughly.

S2: Add ~50 mLs of Milli-Q water to a 100 mL Class A volumetric flask. Re-pipette 1 mL conc. HNO_3 and 5 mLs conc. HCl into the flask. Using Eppendorf pipettes, add 1.0 mL each of RFW-ICPT-STD-2A, RFW-ICPT-STD-2B, and RFW-ICPT-STD-3. Dilute to volume with Milli-Q water and mix thoroughly.

S2A: Add ~50 mLs of Milli-Q water to a 100 mL Class A volumetric flask. Re-pipette 1 mL conc. HNO_3 and 5 mLs conc. HCl into the flask. Using Eppendorf pipettes, add 0.4 mLs each of RFW-ICPT-STD-2A, RFW-ICPT-STD-2B, and RFW-ICPT-STD-3. Dilute to volume with Milli-Q water and mix thoroughly.

S2B: Add ~50 mLs of Milli-Q water to a 100 mL Class A volumetric flask. Re-pipette 1 mL conc. HNO_3 and 5 mLs conc. HCl into the flask. Using Eppendorf pipettes, add 0.5 mLs of RFW-ICPT-STD-2A, RFW-ICPT-STD-2B, and RFW-ICPT-STD-3. Dilute to volume with Milli-Q water and mix thoroughly.

5.2.2 QC Solutions (Refer to Appendix B for element concentrations.)

Prepared with Milli-Q water that has been acidified with 1% HNO_3 and 5% HCl. All QC Solutions are recorded in the intermediate standard traceability logbook. The QC Solutions are prepared as follows:

1. **Initial Calibration Verification (ICV)**

Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. Add 10 mLs conc. HNO_3 and 50 mLs conc. HCl. Add 8 mLs each of CCV Soln. A, CCV Soln. A1, CCV Soln. B and the following:

- 1.84 mLs of 10,000 ug/mL Ca
- 1.6 mLs of 10,000 ug/mL Na, Fe
- 1.68 mLs of 10,000 ug/mL Mg
- 3.6 mLs of 10,000 ug/mL K, Al

Dilute to volume with Milli-Q water and mix thoroughly.

2. **Continuing Calibration Verification (CCV)**

Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. Add 10 mLs conc. HNO_3 and 50 mLs conc. HCl. Add 10 mLs each of CCV Soln. A, CCV Soln. A1, CCV Soln. B and the following:

- 2.3 mLs of 10,000 ug/mL Ca
- 2.0 mLs of 10,000 ug/mL Na, Fe
- 2.1 mLs of 10,000 ug/mL Mg
- 4.5 mLs of 10,000 ug/mL K, Al

Dilute to volume with Milli-Q water and mix thoroughly.

3. **CRI [Contract Required Detection Limit (CRDL) Standard for ICAP]**

(Refer to Appendix B for element concentrations.)

Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. Add 10 mLs conc. HNO_3 and 50 mLs conc. HCl to the flask. Add the following:

- 20 uL each of 10,000 ug/mL Fe
- 40 uL of 10,000 ug/mL Al
- 100 uL of CRI-CRA-1
- 200 uL each of 1000 ug/mL B, Bi, Li, Mo, Si, Sn, Sr, Ti, CRI-CRA-2, CRI-CRA-3
- 400 uL of 1000 ug/mL Ba
- 1 mL each of 10,000 ug/mL Ca, K, Mg, Na

Dilute to volume with Milli-Q water and mix thoroughly.

4. **Interferent Check Standard (ICSA)**

(Refer to Appendix B for element concentrations.)

Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. Add 10 mLs conc. HNO_3 and 50 mLs conc. HCl. Add 100 mLs of CLP Interferent A Solution. Dilute to volume with Milli-Q water and mix the solution thoroughly.

5. **Interferent Check Standard (ICSAB)**

(Refer to Appendix B for element concentrations.)

Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. Add 10 mLs conc. HNO_3 and 50 mLs HCl. Add 100 mLs of CLP Interferent A Solution, 10 mLs of CLPP-ICS-B4. Bring up to volume with Milli-Q water and mix thoroughly.

6.0 CALIBRATION (NON-DAILY)

6.1 Linear Range Analysis Standard (LRS)

LRS calibration is performed quarterly that covers the anticipated range of measurement. This is used to verify linearity and document the upper limit of the calibration range for each element. At least one of the calibration standards will be at or near the reporting limit. The calibration curve generated must have a correlation coefficient of $\geq$ 0.995 in order to consider the responses linear over that range. All samples found to be above the ICAP linear range are diluted and re-analyzed until the concentration falls within the instruments linear range.

6.2 Inter-Element Correction (IEC)

Correction factors for spectral interference due to Al, Ca, Fe, and Mg will be determined at least annually for all wavelengths used for each analyte reported or any time the ICAP is adjusted in any way that may affect the IECs. Correction factors for spectral interferences other than Al, Ca, Fe, and Mg are recommended and are performed as needed and documented with the instrument records.

7.0 PROCEDURE

7.1 Quality Control Checks

The following section summarize the quality control (QC) samples associated with ICAP analysis.

QC Sample	Frequency	Control Limit [1]
Method Blank (MB)	1 per 20 samples	$\leq$ Reporting Limit
Lab Control Sample (LCS) [2]	1 per 20 samples	80 – 120 %
Matrix Spike (MS) [3]	1 per 20 samples	75 – 125 %
MS Duplicate (MSD) [3]	1 per 20 samples	75 – 125 %; 20 RPD
Duplicates (MD) [4]	1 per 20 samples	20 RPD
Serial Dilution (5x) [5]	1 per 20 samples	$\pm$ 10% of the original result

[1] Refer to Section 8 for additional details.
[2] LCS Duplicate (LCD) is performed only when requested by the client or project.
[3] If sample concentration is $\leq$ 4X spike level, 75-125%; if sample concentration is > 4X spike level, no control range. If TCLP matrix spike is < 50%, Standard Addition must be performed.
[4] If $\geq$ 5X reporting limit, 20 RPD; if < 5X reporting limit $\pm$ reporting limit; if < reporting limit no control range.
[5] If the analyte concentration is >10X the MDL, results should agree within $\pm$10% of the original sample result.

7.2 Sample Preservation and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Holding Time[1]	Preservation	Reference
Waters	180 days	HNO_3, pH < 2; Cool 4 ± 2°C	40 CFR Part 136.3
Soils	180 days	Cool 4 ± 2°C	N/A

[1] Inclusive of digestion and analysis.

7.3 Sample Preparation

The most commonly used digestion procedures are SW-846 Methods 3010A (waters) and 3050B (soils). Refer to USP-3000 for details on sample digestion. The samples are received in the metals laboratory as 25, 50 or 100 mL final volumes.

7.4 Calibration / Standardization

7.4.1 Instrument Set Up

Set up the instrument with the proper operating conditions as provided by TJA. The instrument must be allowed to become thermally stable (usually requiring ~1-hour) prior to profiling and calibration. The instrument is profiled using a 1 ppm Arsenic standard (S1) by aspiration and selecting the automatic profile feature from the TJA software. The peak position reading should be within +/- 0.1. If the reading is acceptable, record the peak area in the logbook & rinse. If the reading is > +/- 0.1, set the micrometer to the adjusted vernier position given by the instrument and profile again to verify. Record the peak area in the logbook and rinse. The instrument is now ready to calibrate.

7.4.2 Standardization

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within the linear range of the instrument.

The instrument is standardized using a calibration blank and 3 calibration standards, which consist of 6 multi-element solutions. The results are given in intensities. Minimum requirement is a blank and a standard.

Standard	Frequency	Control Limit
Calibration Curve	Initially	Corr. Coeff. $\geq$ 0.995
High Standards (S1, S2)	After the Calibration Curve	$\pm$ 5% of the Known Conc.
Initial Cal. Verif. (ICV)	After the Calibration Curve	$\pm$ 10% of the Known Conc.
Initial. Cal. Blank (ICB)	After the ICV	$\leq$ Reporting Limit
CRI	Daily, every 8 hrs. thereafter	None Required
ICSA / ICSB	Daily, every 8 hrs. thereafter	$\pm$ 20% of the Known Conc.
Cont. Cal. Verif. (CCV)	Every 10 reading; End of each run	$\pm$ 10% of the Known Conc.
Cont. Cal. Blank (CCB)	Every 10 readings; End of each run	$\leq$ Reporting Limit

7.5 Preventive Maintenance

The required preventive maintenance is listed in the preventive maintenance logbooks which are kept at the instruments. All maintenance is recorded in these logbooks along with the date and the signature of the analyst performing the maintenance. The instruments are under a full service contract with the manufacturer for all major repairs.

7.5.1 Daily Maintenance

Includes changing the pump tubing for consistent sample uptake and a visible check of the waste container to make sure that it doesn't overflow.

7.5.2 Weekly Maintenance

Includes checking the air filters on the back of the instrument for excessive dust buildup, and checking the tip of the torch for excessive buildup of material.

7.5.3 Monthly Maintenance

Includes cleaning and checking the water re-circulator for proper fluid level, cleaning the spray chamber.

7.6 Sample Analysis

7.6.1 Analytical Run

After the instrument is standardized (Section 7.4.2), an analytical run is initiated. The first run of the day would proceed as follows:

- S1,S2 Reanalysis of calibration standard as a sample
- ICV Initial Calibration Verification
- ICB Initial Calibration Blank

- CRI Spiked Blank Sample
- ICSA Interferent Check Standard A
- ICSB Interferent Check Standard B
- CCV Continuing Calibration Verification
- CCB Continuing Calibration Blank
- MB Method Blank
- LCS Laboratory Control Sample
- Sample 1
- Sample 1 Matrix Duplicate (MD)
- Sample 1 Matrix Spike (MS)
- Sample 1 Matrix Spike Duplicate (MSD)
- Sample 1 Serial Dilution (L)
- Sample 2
- •
- •
- Sample X (10)
- CCV Continuing Calibration Verification
- CCB Continuing Calibration Blank

If the CCV and CCB results are acceptable, the run may continue without restandardization. If any of the post-run QC is out of control, or close to being out of control, the instrument is restandardized before analyzing the next batch. Any samples with elements associated with an out of control CCV or CCB will be reanalyzed.

7.7 Documentation

7.7.1 Instrument Run-Log

The analysis of samples and standards is documented within the instrument run log and supported by the instrument print-out. The runlog must be completed for each days analysis. An example of a runlog page appears in Appendix C.

7.7.2 Traceability of Standards

Custom made and single element stock standard solution which are traceable to NIST or EPA are purchased. Upon receipt, each standard is entered into LabNet and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, date of verification and the initials of the recording analyst are also entered.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. The analyst and a trained data reviewer perform the reviews according to the criteria established on the data review checklist (Refer to Appendix D). Upon the first 100% review, the checklist is initialed and dated as reviewed. The package, with its review sheet, comments and any Corrective Action Reports (CARs) are submitted to the section manager, unit supervisor or peer reviewer for a second review. Once again, the checklist is initialed and dated by the second reviewer. The completed data review form remains on file with the original data.

8.0 QUALITY CONTROL

8.1 QC Summary

NOTE: The following laboratory acceptance criteria are set at default control limits. Statistical limits are generated on an annual basis from cumulative LCS data and can be implemented when specified by the client, contract, or QAP.

8.1.1 Method Blank (MB)

At least one MB and one LCS will be included in each digestion batch of 20 samples. Regardless of the matrix being processed, the LCS and MB will be in an aqueous media. The MBs are analyzed to determine if contaminants are being introduced into the sample via the sample preparation procedures.

8.1.2 Laboratory Control Sample (LCS)

The LCS is analyzed to determine the accuracy of the digestion process.

Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be within $\pm$20% of the known concentration. If the LCS results are outside these control limits, all samples in the preparation set must be redigested and reanalyzed. Refer to Appendix E for element concentrations.

8.1.3 Matrix Duplicate (MD)

A duplicate sample will be prepared at a frequency of 5% (1 in 20 samples). A 20 RPD is set as the acceptance limits.

8.1.4 Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

The MS / MSD will be prepared at a frequency of 5% (1 in 20 samples). The recovery must be within 75 - 125%. (Exception allowed if the sample concentration exceeds 4 times the spike added concentration.)

TCLP - If the MS recovery is <50% and the concentration does not exceed the regulatory limit or the sample concentration is within 20% of the regulation level, the Method of Standard Addition (MSA) is required. Three aliquots of the sample are spiked at 50%, 100% and 150% of the sample concentration or, if the sample concentration is < RL, the MSA is at 50%, 100% and 150% of the MS level. The data is subjected to linear regression whereas the concentration of the unknown is the x-intercept and the correlation coefficient value must be $\geq$ 0.995.

8.1.5 Serial Dilution

A Serial Dilution (5X) will be prepared from the digestate at a frequency of 5% (1 in 20 samples). If the concentration is >10 times the MDL, results should agree within +/- 10% of the original results.

8.2 Corrective Action

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out of control situation may be caused by more than one variable. The analyst should seek the assistance of his/her section manager, supervisor, QA personnel, or other experienced staff if he/she are uncertain of the cause of the out of control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the section manager, supervisor, or QA personnel.

Listed below are steps that must be taken when an out of control situation occurs:
- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on a CAR;
- document on the CAR that an in control has been achieved; and
- receive approval (signature) of the section manager, supervisor, or QA personnel prior to the release of any analytical data associated with the problem.

Suggested actions to specific out of control situations:

8.2.1 Calibration Curve
- reanalyze the standard curve;
- prepare a new stock and/or working standards;
- check the reagents/solutions and prepare fresh if necessary.

8.2.2 Initial Calibration Verification (ICV)
- repeat the ICV to verify proper preparation;
- prepare a new ICV from original stock;
- recalibrate with a new standard curve;
- prepare a new stock and/or working standards;

- check the reagents/solutions and prepare fresh if necessary.

8.2.3 Initial Calibration Blank (ICB)

- prepare a new ICB to verify proper preparation;
- verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability;
- determine the source of contamination by process of elimination, carryover from a previous analysis or reagent contamination and correct the problem;
- check the reagents/solutions and prepare fresh if necessary;
- correct for any contamination and reanalyze the ICB and any associated samples.

8.2.4 Laboratory Control Standards (LCS)

If the LCS is low:

- *reanalyze the LCS and all samples in the set for the failed analyte(s) to confirm that it is out of control.*
- If continued out of control, redigest and reanalyze the set.
- Write a CAR.

If the LCS is high:

- reanalyze the LCS and all samples in the set for the failed analyte(s) to confirm that it is out of control.
- check for contamination of reagents, LCS stock solution, or in the preparation area;
- correct for contamination, redigest and re-analyze the set;
- Write a CAR.

8.2.5 Laboratory Control Standard Duplicate (LCD)

- Performed on a project-by-project basis and project-specific corrective actions will be applied.

8.2.6 Method Blank (MB)

- reanalyze the MB to verify that it is beyond the reporting limit;
- determine the source of contamination;
- determine if a high value is due to contamination;
- check for contamination of reagents or in the preparation area;
- correct for contamination, reanalyze the set;
- in the extreme case where all samples in the set are at least 10x > the MB or < RL, reanalysis will not be required; however, a CAR will be written and approved by the supervisor or section manager

8.2.7 Matrix Duplicate (MD)

- a CAR will be written and approved by the supervisor or section manager.

8.2.8 Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

- a CAR will be written and approved by the supervisor or section manager.

8.2.9 Serial Dilution (L)

- prepare a new serial dilution to verify proper preparation;
- a CAR will be written and approved by the supervisor or section manager.

8.2.10 Continuing Calibration Verification (CCV)

- repeat the CCV to verify proper preparation;
- prepare a new CCV from the original stock;
- check for instrument base-line drift or a change in one or more of the reagents;
- check the reagents/solutions and prepare fresh if necessary;
- recalibrate with a new standard curve and repeat all samples since the previous in control CCV;
- never dispose of any samples until you are sure that all QC are within the control limits.

8.2.11 Continuing Calibration Blank (CCB)

- check reagents/solutions to verify proper preparation and prepare fresh if necessary;
- verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc., to achieve stability;
- correct for any contamination (carryover from a previous analysis or reagent contamination) and reanalyze the CCB and any associated samples;
- never dispose of any samples until you are sure that all QC are within the control limits.

8.2.12 Additional Corrective Actions

1. If any of the ICV, ICB, ISA, ISB, CCV or CCB results are out of control for any element, the instrument is restandardized and the samples associated with the out of control elements are reanalyzed.

2. If the MB or LCS are out of control for any element, the samples are redigested. An exception is if the sample concentrations are $\geq$ 10X the MB contamination or < RL. In this case, the results are reported as is.

3. If any of the MD or MS/MSD results are out of control, the client is notified of the poor results via a case narrative that is sent with the data report.

4. CARs are completed by the analyst performing the analysis. The forms are then reviewed and signed by the supervisor or section manager. The signed forms are filed with the original data and a copy is kept on file in the Metals Department.

9.0 DATA ANALYSIS AND CALCULATIONS

The sample results are stored in a data file on the desktop computer. The data is transferred over to LabNet and edited there. This system helps to eliminate transcription errors, since data is not entered by hand.

9.1 Accuracy

9.1.1 ICV / CCV, LCS

$$\% \text{ Recovery} = \frac{\text{observed concentration}}{\text{actual concentration}} \times 100$$

9.1.2 MS / MSD

$$\% \text{ Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

9.2 Precision

9.2.1 Matrix Duplicate (MD)

$$\text{RPD} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

9.3 Concentration

$$\text{mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mLs)

9.4 Dry Weight Reporting

$$= \frac{\text{Final Result}}{\text{Total Solids}} \times 100$$

NOTE: All dry weight corrections are made in LabNet at the time the final report is prepared.

10.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- Waste from this procedure will enter the "Corrosive Wastewater" wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1.0, 7.0 and 8.0.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Table 1:	Element and Reporting Limits
Appendix A:	Standard Stock Solutions
Appendix B:	Stock QC Solutions
Appendix C:	Example: Analysis Run Log
Appendix D:	Example: Data Review Form
Appendix E:	Known Digested Quality Control

Historical File:

Revision 00: 02/11/98
Revision 01: 01/29/99
Revision 02: 03/20/00
Revision 03: 06/29/01
Revision 04: 09/13/02

Reasons for Revision; Revision 04

- Annual Review – No Changes.
- Updated the Health & Safety (3.0) and Waste Disposal (10.0) sections.

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Table 1.

Element and Reporting Limits

Element	ICAP 61E (ICP3) Wavelength (nm)	ICAP 61E (ICP4) Wavelength (nm)	ICAP 61E (ICP5) Wavelength (nm)	Reporting Limits[1]	
				Waters (ug/L)	Soils (mg/kg)
Al	308.2	308.2	308.2	200	20
Sb	206.8	206.8	206.8	20	2
As	189.0	189.0	189.0	10	1
Ba	493.4	493.4	493.4	10	1
Be	313.0	313.0	313.0	4	0.4
Bi	223.0	223.0	N/A	50	5
B	249.6	249.6	249.6	50	5
Ca	317.9	317.9	317.9	100	10
Cd	226.5	226.5	226.5	2	0.2
Cr	267.7	267.7	267.7	10	1
Co	228.6	228.6	228.6	5	0.5
Cu	324.7	324.7	324.7	10	1
Fe	271.4	271.4	271.4	50	5
Pb	220.3	220.3	220.3	5	0.5
Mg	279.0	279.0	279.0	100	10
Mn	257.6	257.6	257.6	10	1.0
Mo	202.0	202.0	202.0	10	1
Ni	231.6	231.6	231.6	10	1
P	214.9	178.2	178.2	50	5
K	766.4	766.4 / 404.7	766.4	500 / 10,000	50 / 1,000
Se	196.0	196.0	196.0	5	0.5
Si	288.1	288.1	288.1	200	20
Ag	328.0	328.0	328.0	5	0.5
Na	330.2	330.2	330.2 / 588.9	1,000	100
Sr	421.5	NA	421.5	5	0.5
Tl	190.8	190.8	190.8	10	1
Sn	189.9	189.9	189.9	20	2
Ti	334.9	337.2	334.9	5	0.5
V	292.4	292.4	292.4	5	0.5
Y[2]	371.0	371.0	371.0	N/A	N/A
Zn	213.8	206.2	206.2	10	1

[1]These are routine Trace ICAP reporting limits (RL). Lower RLs are available and can be used per client request. RLs will vary depending on sample size/volume, dilution factors, dry weight reporting for soils, and changes in MDLs.
[2]Y is used as an internal standard and is introduced continuously to all samples (including standards and QC samples) via the peristaltic pump at an approximate concentration of 5 ppm.

Appendix A.

Standard Stock Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	S1A	S1B	S1	S2A	S2B	S2
Inorganic Ventures	RFW-ICPT-STD-1B	Sb	100	0.4	0.5	1			
		Mo	100	0.4	0.5	1			
		Si	100	0.4	0.5	1			
		Sn	100	0.4	0.5	1			
		Ti	100	0.4	0.5	1			
Inorganic Ventures	RFW-ICPT-STD-1C	Al	1000	4	5	10			
		Fe	1000	4	5	10			
		K	1000	4	5	10			
		Na	1000	4	5	10			
		Li	800	2	4	8			
		Mg	800	2	4	8			
		Ca	400	1.6	2	4			
Inorganic Ventures	RFW-ICPT-STD-1D	As	100	0.4	0.5	1			
		Ba	100	0.4	0.5	1			
		Be	100	0.4	0.5	1			
		Bi	100	0.4	0.5	1			
		B	100	0.4	0.5	1			
		Cd	100	0.4	0.5	1			
		Cr	100	0.4	0.5	1			
		Cu	100	0.4	0.5	1			
		Pb	100	0.4	0.5	1			
		Ni	100	0.4	0.5	1			
		Se	100	0.4	0.5	1			
		Ag	100	0.4	0.5	1			
		Sr	100	0.4	0.5	1			
		Tl	100	0.4	0.5	1			
		Zn	100	0.4	0.5	1			
Inorganic Ventures	RFW-ICPT-STD-2A	Al	10,000				40	50	100
		K	10,000				40	50	100
Inorganic Ventures	RFW-ICPT-STD-2B	Ca	5000				20	25	50
		Fe	5000				20	25	50
		Mg	5000				20	25	50
		Na	5000				20	25	50
Inorganic Ventures	RFW-ICPT-STD-3	Pb	2000				8	10	20
		Mn	1000				4	5	10
		V	1000				4	5	10

Appendix B.

Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICV (mg/L)	CCV (mg/L)
High Purity	CCV Solution A	As	50	0.4	0.5
		B	50	0.4	0.5
		Ba	50	0.4	0.5
		Be	50	0.4	0.5
		Bi	50	0.4	0.5
		Cd	50	0.4	0.5
		Co	50	0.4	0.5
		Cr	50	0.4	0.5
		Cu	50	0.4	0.5
		Ni	50	0.4	0.5
		Pb	50	0.4	0.5
		Se	50	0.4	0.5
		Fe	500	20	25
		Mn	500	4	5
		V	500	4	5
		Tl	50	0.4	0.5
		Zn	50	0.4	0.5
		Sr	50	0.4	0.5
High Purity	CCV Solution A2	Ca	200	20	25
		Li	400	---	---
		Na	500	20	25
		Al	500	40	50
		Mg	400	20	25
		K	500	40	50
High Purity	CCV Solution B	Ag	50	0.4	0.5
		Sb	50	0.4	0.5
		Mo	50	0.4	0.5
		Si	50	0.4	0.5
		Sn	50	0.4	0.5
		Ti	50	0.4	0.5
Ultra	Single Elements	Al	10,000	40	50
		Ca	10,000	20	25
	* spiked on top	Fe	10,000	20	25
	of custom mixes.	Na	10,000	20	25
		K	10,000	40	50
		Mg	10,000	20	25

Appendix B.
(continued)
Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	CRI Conc. (mg/L)
Inorganic Ventures	CRI-CRA-1	Be	100	0.01
		Cr	200	0.02
		Co	1000	0.10
		Cu	500	0.05
		Mn	300	0.03
		Ni	800	0.08
		Ag	200	0.02
		V	1000	0.10
		Zn	400	0.04
Inorganic Ventures	CRI-CRA-2	Sb	600	0.12
Inorganic Ventures	CRI-CRA-3	As	100	0.02
		Cd	50	0.01
		Pb	30	0.006
		Se	50	0.01
		Tl	100	0.02
Inorganic Ventures	Calcium	Ca	10,000	10
	Potassium	K	10,000	10
	Magnesium	Mg	10,000	10
	Sodium	Na	10,000	10
	Iron	Fe	10,000	0.2
	Aluminum	Al	10,000	0.4
	Barium	Ba	1,000	0.4
	Boron	B	1,000	0.2
	Bismuth	Bi	1,000	0.2
	Molybdenum	Mo	1,000	0.2
	Silicon	Si	1,000	0.2
	Tin	Sn	1,000	0.2
	Strontium	Sr	1,000	0.2
	Titanium	Ti	1,000	0.2

Appendix B.
(continued)
Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICSA Conc. (mg/L)
Inorganic Ventures	CLP Interferents "A" Solution	Al	5000	500
		Ca	5000	500
		Mg	5000	500
		Fe	2000	200
				ICSB Conc. (mg/L)
Inorganic Ventures	CLP Interferent A Solution	Al	5000	500
		Ca	5000	500
		Mg	5000	500
		Fe	2000	200
Inorganic Ventures	CLPP-ICS-B4	Cd	100	1
		Ni	100	1
		Zn	100	1
		Sb	60	0.6
		Ba	50	0.5
		Be	50	0.5
		Co	50	0.5
		Cr	50	0.5
		Cu	50	0.5
		Mn	50	0.5
		V	50	0.5
		Ag	20	0.2
		As,Tl	10	0.1
		Pb,Se	5	0.05

Appendix C.

Example: Analysis Runlog

STL Chicago

TJA Trace ICAP (61E) Analysis Log – ICP5

Page No._______________

Date	Initials	File Name	Dig. Set	Int. Std	Sample Nos.	Parameters	Comments
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			
				As =			
				Y =			

Reviewed by:______________________________ Date:____________________

Appendix D.

Example: Data Review Checklist

STL Chicago
ICAP Metals Data Review Checklist

Instrument ID: **ICP 3** **ICP 4** **ICP 5** **Filename:**______________________

Analyst Initial(s):______________________ **LabNet Batch No.:**______________________

Copies: ______________________ ______________________

______________________ ______________________

______________________ ______________________

______________________ ______________________

______________________ ______________________

****

QC Type: a. CLP b. Standard c. TCLP d. Drinking Waters e. Solubles

I. Calibration:

Analyst	Reviewer	
☐	☐	1. Verification of standard traceability and expiration (daily).
		2. Calibration is clearly documented:
☐	☐	a. Instrument is calibrated using a Blank and three Calibration Standards. The correlation coefficient must be $\geq$0.995.
☐	☐	b. Reanalysis of the top calibration standard as a sample. Control limits are 95 - 105%. (Run once daily prior to sample analysis).
		3. Calibration Verification: (10% Frequency):
☐	☐	a. ICV/CCV: **Std./CLP** – Recovery 90-110% **EPA 200.7 (ICV)** – Recovery 95-105%
☐	☐	b. ICB/CCB: **Std. QC:** < RL; **CLP QC:** < CRDL; **SW-846 QC:** < 3x IDL.
		4. **CLP QC:** An Initial & Final for each sample analysis run:
☐	☐	a. CRI - 2x CRDL; No Limit Set
☐	☐	b. ISA/ISAB - 80-120% Recovery
		5. **Std. QC:** Analyzed at the beginning of the day and every 8 hours thereafter:
☐	☐	a. CRI: 2x CRDL; No Limit Set
☐	☐	b. ISA/ISAB: 80-120% Recovery
		Refer to Run #:______________________

Note: **CLP QC** requires the use of the IDL for calculating % Recoveries and Reporting Limits.
Standard QC requires the use of the RL for calculating % Recoveries and Reporting Limits.

II. Sample Analysis:

Analyst	Reviewer	
		1. Each Prep Batch consists of a maximum of 20 samples of a similar matrix:
☐	☐	a. Prep Batches must be clearly identified
☐	☐	b. 1 Prep Blank **CLP** - < CRDL; **Std. QC** - < RL **TCLP** - < TCLP Reporting Limit
☐	☐	c. 1 LCS **Std./CLP** - 80-120% Rec.; **EPA 200.7** - 85-115% Rec.
☐	☐	d. 1 Duplicate **Std.** - RPD or RSD limits are 20%; ***Unless*** the sample conc. is <5x RL then $\pm$ RL applies; for CLP $\pm$ CRDL applies. **EPA 200.7** - 10% Frequency
☐	☐	e. 1 Matrix Spike **Std./CLP** - 75-125% Rec.; ***Unless*** the sample conc. exceeds the spike conc. by 4x; **EPA 200.7** - 70-130% Rec.; 10% Frequency
☐	☐	f. Analytical MS **TCLP** - >50% (MSA performed if <50% recovery)
☐	☐	g. Serial Dilution 1 per 20 samples; 10% Difference Limit
☐	☐	h. A post-digestion spike (PMS) must be performed for CLP and 200.7 if the above limits are not met, (CLP - except for Ag, Na, Ca, K, and Mg for waters and soils, and Al and Fe for soils only).
☐	☐	i. Turbidity Checked: **EPA 200.7 Drinking Water**

STL Chicago
ICAP Metals Data Review Checklist

II. Sample Analysis (continued):

Analyst	Reviewer	
		2. A Corrective Action Report (CAR) must be written for any out of control situations, clearly stating the problem and action to be taken:
☐	☐	a. CAR included with original data run
☐	☐	b. CAR with corrective action results included with the corrective action run.

III. Data Documentation

Analyst	Reviewer	
		1. Raw Data:
☐	☐	a. Unused data is clearly identified.
☐	☐	b. All crossed out data is initialed and dated.
☐	☐	c. Out of control QC is clearly identified.
☐	☐	d. Any data that has a tick (S, I, H or L) is commented on with appropriate action taken.
☐	☐	e. The first page of the run must have the filename; instrument; and analyst's signature

		2. Run Log:
☐	☐	a. Unused data is clearly identified.
☐	☐	b. All cross outs are initialed and dated.
☐	☐	c. Analyst's Signature is required.

		3. LabNet:
☐	☐	a. Worksheet and data pages are printed.
☐	☐	b. Unused data is clearly identified.
☐	☐	c. All cross-outs are initialed and dated.
☐	☐	d. First page must have the filename, instrument identification; analyst signature.
☐	☐	e. Samples needing copying are clearly marked.
☐	☐	f. Label Sample ID with the LabNet Batch their in.

III. Miscellaneous

Analyst	Reviewer	
☐	☐	1. Is Sample Prep Linked?
☐	☐	2. Is TCLP Linked? (Shift F9 from the start page)
☐	☐	3. Did all dilutions carry over for MD, MS, MSD (where applicable)?
☐	☐	4. Did all prep and analysis matrices match up?

Comments:

____

____

____

Analyst Signature: ____________________________ Date:

Reviewer Signature: ____________________________ Date:

Appendix E.

Known Digested QC Values (mg/L)

Element	LCS/Spike	TCLP Spike
Al	2	-
Sb	0.5	-
As	0.1	5
Ba	2	100
Be	0.05	-
Bi	0.5	-
B	1	-
Cd	0.05	1
Ca	10	-
Cr	0.2	5
Co	0.5	-
Cu	0.25	0.25
Fe	1	-
Pb	0.10	5
Mg	10	-
Mn	0.5	-
Mo	1	-
Ni	0.5	0.5
P	0.5	-
K	10	-
Se	0.10	1
Si	5	-
Ag	0.05	1
Na	10	-
Sr	1	-
Tl	0.10	-
Sn	1	-
Ti	1	-
V	0.5	-
Zn	0.5	-

<u>Default Control Limits</u>
LCS: 80 - 120%
Spike: 75 - 125%
TCLP Spike: >50%

TITLE: Metals Analysis Mercury by EPA Methods 245.1/245.5; SW-846 7470A/7471A; and U.S. EPA CLP Document No. ILM04.0

Updated by:	Signature:	Date:
George Klee Mercury Analyst	George O Klee	9/18/02

Approved by:	Signature:	Date:
Mani S. Iyer Section Manager, Metals Dept.	Mani S Iyer	9/24/02
David L. Kaczka Env. Health & Safety Coor.	DLKzK	9/16/02
Terese A. Preston Quality Manager	Terese A. Preston	9/16/02

CONTROLLED DISTRIBUTION

COPY # : Uncontrolled

ISSUED TO : Mark Densmore, Secor
Re: UTC Proposal

Full Signature Approvals Are Kept on File
with STL's QA Standard Practice Records

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the digestion and analytical procedure for the determination of the mercury concentration in aqueous and non-aqueous media. This SOP was written using EPA 600/4-79-020 Methods 245.1 and 245.5; SW-846, 3rd Edition, Methods 7470A/7471A; and U.S. EPA CLP Document No. ILM04.0 as references.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

1.1.2 Instrument Detection Limits

Instrument Detection Limits (IDLs) are performed quarterly for each element by the metals laboratory for each instrument as specified in CLP. These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined.

NOTE: The annual MDL may be used in lieu of one of the semi-annual IDL sets, providing required reporting limits are achieved.

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. Because of the high level of quantitative error associated with determinations at the level of the MDL, the laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately

3-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement.

Matrix	Reporting Limit [1]	CRDL [2]
Water	0.2 ug/L	0.2 ug/L
Soil	0.033 mg/kg	0.1 mg/kg

[1] Reporting Limit is used for EPA Method 245.1 and SW-846 7470A/7471A. Reporting Limits may vary depending on sample volume/size, dilution factors, and changes in the MDL.
[2] CRDL (Contract Required Detection Limit) is used for U.S. EPA CLP ILM04.0.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM, Revision 02).

1.2 Summary of Method

This flameless cold vapor AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and swept from solution and passed through a cell of a double beam AA. Absorbance is a function of mercury concentration.

2.0 INTERFERENCES

- Chloride, sulfide and certain volatile organic materials.

3.0 SAFETY

- Employees will adhere to the practices and policies in the STL Corporate Safety Manual (CSM) and will read the MSDSs for the materials used in this method before handling or using the material.
- As always, general laboratory safety practices should always be followed
- The standards contains potentially harmful levels of mercury. Care should be taken to avoid contact with the stock solutions. Wash hands well if contacted.

4.0 EQUIPMENT AND SUPPLIES

- 2 – Leeman Labs Model PS200 Automated Mercury Analyzer
- Class A volumetric glassware
- Eppendorf pipettes

5.0 REAGENTS AND STANDARDS

5.1 Reagents

5.1.1 Miscellaneous Reagents

- Hydrochloric Acid [HCl], Concentrated
- Nitric Acid [HNO_3], Concentrated
- Sulfuric Acid [H_2SO_4], Concentrated
- Deionized (DI) Water, Type II

5.1.2 Sodium Chloride-Hydroxylamine Hydrochloride Solution

Dissolve 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride in sufficient DI water to make 2-liters of solution.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.1.3 Stannous Chloride Solution

Dissolve 100 g of stannous chloride in 10% hydrochloric acid to make 1-liter of solution.

- Life of Reagent: 1 Month
- Storage Requirements: None

5.1.4 Potassium Permanganate, 5%

Dissolve 175 g of potassium permanganate into 3.5-liters of DI water.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.1.5 Potassium Persulfate, 5%

Dissolve 175 g of potassium persulfate into 3,500 mLs of DI water.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.2 Standards All standards are prepared in Class A volumetric flasks.

5.2.1 Standard Stock Solution I; 1,000 ppm

A 1,000 ppm concentrated mercury standard is purchased from an outside supplier.

- Life of Standard: 1 Year
- Storage Requirements: None

5.2.2 Working Standard Solution I; 100 ppb

To a 1.0 L volumetric flask filled with ~800 mLs DI water, transfer 100 uLs of Stock Solution I to the flask using a 100 uL Eppendorf pipette. Add 2.5 mLs conc. nitric acid as a preservative. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking Matrix Spikes, CRAs & the Standard Curve.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.2.1 Working Standard Solution IA; 25 ppb

To a 100 mL volumetric flask filled with ~80 mLs DI water, transfer 25 uLs of Working Standard Solution I (Item 5.2.2) to the flask using an Eppendorf pipette. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking Matrix Spikes, CRAs & the Standard Curve in the **Hot Block Digester**

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.3 Standard Stock Solution II; 1,000 ppm

Purchased from an outside supplier as a 1,000 ppm solution and is from an alternate source than that of Standard Stock Solution I (Rgt. 5.2.1).

- Life of Standard: 1 Year
- Storage Requirements: None

5.2.4 Working Standard Solution II; 200 ppb

To a 1.0 L volumetric flask filled with ~800 mLs DI water, add 2.5 mLs concentrated nitric acid (as a preservative) and 200 uLs of Standard Stock Solution II to the flask (using a 200 uL Eppendorf pipette). Dilute to volume with DI water and invert several times to mix.

*For use in spiking the ICV/CCV and LCS.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.4.1 Working Standard Solution IIA; 50 ppb

To a 100 mL volumetric flask filled with ~80 mLs DI water, add 25 uLs of Working Standard Solution II (Item 5.2.4) to the flask using an Eppendorf pipette. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking the ICV/CCV and LCS in the **Hot Block Digester**

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.5 Working Standards for Mercury in Water

Standard (ug/L)	mLs of Working Soln. I or IA	Final Volume (mLs) Water Bath	Final Volume (mLs) Hot Block
Blank	0.0	100	25
0.2	0.2	100	25
0.5	0.5	100	25
1.0	1.0	100	25
3.0	3.0	100	25
5.0	5.0	100	25
CRA (0.2 ug/L)	0.2	100	25
Matrix Spike (1.0 ug/L)	1.0	100	25

Standard (ug/L)	mLs of Working Soln. II or IIA	Final Volume (mLs) Water Bath	Final Volume (mLs) Hot Block
Init. Cal. Verif. (ICV) (2.0 ug/L)	1.0	100	25
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.5	100	25
Lab Control Sample (LCS) (2.0 ug/L)	1.0	100	25

CLP Standard (ug/L)	mLs of Working Soln. II or IIA	Final Volume (mLs) Water Bath	Final Volume (mLs) Hot Block
Init. Cal. Verif (ICV) (2.0 ug/L)	1.0	100	25
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.5	100	25

NOTE: ILM04.0 requires the ICV and CCV to be at different levels.

6.0 CALIBRATION (NON-DAILY)

All calibration procedures are performed on a daily basis. Refer to Section 7.4 for details.

7.0 PROCEDURE

7.1 Quality Control Checks

The following Quality Control samples are performed with each batch of samples. Refer to Section 8.0 for additional details.

QC Sample	Frequency[1]	Control Limits
Method Blank (MB)	1 in 20 samples	• < Reporting Limit (EPA / SW-846) • < CRDL (CLP)
LCS	1 in 20 samples	• 80-120% Recovery (EPA / SW-846 / CLP) • 85-115% Recovery (EPA 245.1 Only)
Matrix Duplicate (MD)[2]	1 in 20 samples	• 20 RPD unless the sample conc. is <5x RL, then ± RL. (EPA / SW-846) • 20 RPD unless the sample conc. is <5x CRDL, then ± CRDL. (CLP)
Matrix Spike (MS) MS Duplicate (MSD)[2]	1 in 20 samples	• 75 – 125% Recovery unless the sample concentration > spike level by 4x (EPA / SW-846 / CLP) • 85 – 115% Recovery (EPA 245.1) • > 50% Recovery; if <50% Recovery, Method of Standard Additions (MSA) is required (TCLP)

[1] Drinking waters by EPA 245.1 and CLP analyses are analyzed at a frequency of 1 in 10 samples.
[2] The sample selection for MS/MSD or MS/MD, where appropriate, are rotated among client samples so that various matrix problems may be noted and/or addressed. MD's are performed only when requested by the client/project/contract. The MS/MSD are the routinely performed matrix QC indicators.

7.2 Sample Preservation and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Listed below are the holding times and preservations for the referenced programs.

Program	Preservation [1]	Holding Time [2]
SDWA	pH < 2, Cool 4 ± 2°C	28 days VTS [3]
CWA	pH < 2, Cool 4 ± 2°C	28 days VTS
RCRA	pH < 2, Cool 4 ± 2°C	28 days VTS
CLP	pH < 2, Cool 4 ± 2°C	26 days VTSR [4]

[1] Waters are preserved with nitric acid at pH <2; Soils are preserved at Cool 4 ± 2°C.
[2] Holding times include digestion and analysis.
[3] VTS: Verified Time of Sampling.
[4] VTSR: Verified Time of Sample Receipt.

7.3 Sample Preparation

7.3.1 Mercury Water Digestion Procedure - EPA Method 245.1 / CLP ILM04.0

Item	Full Scale (Water Bath)	Hot Block
Sample Volume	100 mLs	25 mLs
Reaction Vessel	BOD Bottle, 300 mLs	Sample Vials, 50 mLs
Sulfuric Acid (conc.)	5 mLs	1.25 mLs
Nitric Acid (conc.)	2.5 mLs	0.625 mLs
Potassium Permanganate, 5% Sol. (W/V)	15 mLs	3.75 mLs
Potassium Persulfate, 5% Sol. (W/V)	8 mLs	2 mLs
Preparation	2 hrs. @ 90 - 95°C, Cool	2 hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	6 mLs	1.5 mLs
Total Volume	136.5 mLs	34.125 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. It the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

Proceed with the Stannous Chloride addition.

7.3.2 Mercury Water Digestion Procedure - SW-846 Method 7470A

Item	Full Scale (Water Bath)	Hot Block
Sample Volume	100 mLs	25 mLs
Reaction Vessel	BOD Bottle, 300 mLs	Sample Vials, 50 mLs
Sulfuric Acid (conc.)	5 mLs	1.25 mLs
Nitric Acid (conc.)	2.5 mLs	0.625 mLs
Potassium Permanganate,	15 mLs	3.75 mLs

Item	Full Scale (Water Bath)	Hot Block
5% Sol. (W/V)		
Potassium Persulfate, 5% Sol. (W/V)	8 mLs	2 mLs
Preparation	2 hrs. @ 90-95°C, Cool	2 hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	6 mLs	1.5 mLs
Total Volume	136.5 mLs	34.125 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. It the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

Proceed with the Stannous Chloride addition.

7.3.3 Mercury Soil Digestion Procedure - SW-846 Method 7471A

NOTE: Three aliquots of soils (~0.2 g) are combined and digested as one sample.

Item	Full Scale (Water Bath)
Sample Weight	~ 0.6 – 0.7 grams
Reaction Vessel	BOD Bottle, 300 mLs
DI Water, Type II	5 mLs
Aqua Regia [3:1 HCl (conc.) to HNO_3 conc.)]	5 mLs
Preparation	2 min. @ 90-95°C, Cool
DI Water, Type II	50 mLs
Potassium Permanganate, 5% Sol. (W/V)	15 mLs
Preparation	30 min. @90-95°C, Cool
Hydroxylamine Addition	6 mLs
Total Volume	Dilute to 100 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. It the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

Proceed with the Stannous Chloride addition.

7.3.4 Mercury Soil Digestion Procedure - EPA Method 245.5 / CLP ILM04.0

Item	Full Scale (Water Bath)
Sample weight	0.2 - 0.3 grams
Reaction Vessel	BOD bottle, 300 mLs

Item	Full Scale (Water Bath)
Sulfuric Acid (conc.)	5 mLs
Nitric Acid (conc.)	2.5 mLs
Preparation	2 min. @ 90 -95°C, Cool
DI Water, Type II	50 mLs
Potassium Permanganate, 5% Sol. (W/V)	15 mLs
Potassium Persulfate, 5% Sol. (W/V)	8 mLs
Preparation	30 min. @ 90 - 95°, Cool
Hydroxylamine Addition	6 mLs
Total Volume	Dilute to 100 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. It the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

Proceed with the Stannous Chloride addition.

7.4 Calibration / Standardization

Before the instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within this linear range of the instrument.

Standard	Frequency	Control Limit
Calibration Curve	Initially	Corr. Coeff. $\geq$ 0.995
ICV	After the Calibration Curve	• 90 –110% Recovery (SW-846 / CLP) • 95 – 105% Recovery (EPA)
ICB	After the ICV	• < Reporting Limit (EPA / SW-846) • < CRDL (CLP)
CRA	After ICB	• No established limits.
CCV	Every 10 readings; end of each run	• 90 – 110% Recovery (EPA / CLP) • 80 – 120% Recovery (SW-846)
CCB	Every 10 readings; End of each run	• < Reporting Limit (EPA / SW-846) • < CRDL (CLP)

7.4.1 Calibrating the System

The instrument must be calibrated before samples are analyzed.

To perform a standard EPA (Method 7470) calibration, press the F2 macro key and "Macro:" prompt appears at the top of the, type "CLP3 STOP" and press enter. The calibration routine will begin running. It is assumed that the five standards (0, 0.2, 0.5, 1.0, 3.0, and 5.0 ppb) have been loaded as standards 1 through 6. After the standards run, the check standards will run automatically. CLP3 STOP will accept the calibration. "Macro:" RUNSTD will run standards only.

NOTE: If running ILM04.0 samples, choose "CLP3 STOP" to run ICV from Check Std. 2 (2 ppb) and all CCV's from Check Std. 3 (1 ppb).

To perform a calibration other than a standard EPA procedure, press the STD F6 action key. The Standard screen appears and a "Run standard: 1 2 3 4 5 6 " message is displayed at the bottom of the screen. Enter the number of the standard to be run (1-6) and press enter. A "from replicate: 1 to: _ "message will then be displayed at the bottom of the screen. Enter the first number in the "from replicate:" field and last number in the "to:" field. Press ENTER. The system will run the standards.

NOTE: To stop a procedure at any time, press the Stop F10 action key.

The results of the calibration are automatically stored. To review the results, select CALIBRATION from the Main Menu and then select LINE CALIBRATION to generate a display.

<u>Below are some guidelines for determining whether the results are acceptable:</u>
- Do the %RSD's look acceptable for various concentrations?
- Is the correlation coefficient larger than 0.995?

If the calibration results are acceptable, type A and press ENTER. A "New calibration coefficients stored" message will be displayed at the bottom of the screen and the samples can now begin to be analyzed..

7.4.2 Check Standards

This option allows for the verification that the calibration has not drifted. To check standard concentrations:

- From the Main Menu, select CALIBRATION and then select CHECK STANDARDS. The check standard screen will appear.
- Type 1 for a check standard blank. Enter, in units specified on the standards page, the range of acceptance.
- Type 2 for check standards cup 2. Type the concentration and Enter. Type the percent acceptance and Enter.
- Repeat this for up to seven check standards.
- From Main Menu, select AUTOSAMPLES, then select SETUP and then check Enter the C1 frequency (e.g., 5/EPA protocol)
- Halt: Enter Y if the instrument should halt after an unacceptable check standard. Enter N for an alert only. Macros can be written to automatically recalibrate and rerun samples if check standards fall outside specifications.

7.5 Preventive Maintenance

The instrument requires some routine daily maintenance as well as some scheduled and non-scheduled periodic maintenance. All maintenance will be recorded in the instruments maintenance logbook. The following maintenance schedule lists the various maintenance procedures and when they should be performed. Each of these procedures is described in the following sections.

7.5.1 Maintenance Schedule

Equipment	Schedule
Drying Tube	Must be changed daily.
Pump Tubing	Weekly, or as needed.
Lamp	Replace as needed (avg. 4 mos. - 1 yr.).
Optical Cell	Clean as needed (typically monthly).
Liquid Gas Separator	Replace every 1-3 yrs., as needed.
Internal Tubing	Should not require replacement under normal circumstances.

7.5.2 Packing and Changing the Drying Tube

Under normal use, the drying tube must be changed each morning before analyzing samples. (The drying tube is located on the front panel on the left side of the instrument) Several tubes can be packed at one time and stored in an airtight container for a ready supply.

To pack a tube, plug one end with quartz wool, pour in magnesium perchlorate to fill tube, and plug the other end with quartz wool.

To change a tube, slightly loosen the nuts that hold the tube in at either end and slide the used tube out of the fittings. Slide a fresh tube into the fittings and tighten the fittings with your fingers to make a gas-tight seal.

To clean a tube, remove the quartz wool and the magnesium perchlorate. Either dispose of as a solid waste or dissolve in water and dispose of as a liquid waste. Clean the tube with ordinary laboratory glassware cleaner and dry thoroughly.

7.5.3 Replacing and Conditioning Pump Tubing

Pump Tubing should be replaced weekly or when it shows signs of wear. There are four pump tubes: two for drainage, one for sample, and one for reductant. Each tube is fed through a pump cassette which then clamps onto the pump head. Slide a tube through the plastic clips at the bottom of a cassette until the plastic tab is secure. Hold the tube taut, slide the loaded cassette onto the pump head, and lock the clamp up. Repeat for the remaining tubes, then connect the tubes ends.

For optimal performance, run DI water through new tubes for one hour to exercise them before using them for running samples. To do this, select INSTRUMENT from the Main Menu and then select OPERATION.

The INSTRUMENT:OPERATION screen will appear. Set the Pump Rate flow to the standard rate for 5 mL/min (Type R and M and 5 Enter). Wait for one hour and then connect the tubing to the appropriate fluids.

NOTE: This procedure only needs to be done once, when the tubes are new and unused.

7.5.4 Replacing the Lamp

The mercury lamp has a life of about 2000 hours, between four months and a year of use. The lamp needs to be replaced if the relative absorbance of a standard has changed significantly while the optical cell is clean. If the lamp is suspected, it is faster to replace the lamp and recalibrate than to clean the optical cell.

NOTE: Before installation, clean the new lamp quartz with methanol and wipe it dry. Do not get finger prints on the lamp and do not face the printing on the lamp toward the optical cell.

- Turn off the lamp (press the blue button on the front of the instrument).
- Remove the front panel of the instrument (lift up and out).
- Remove the optical assembly.
- Remove the two screws on the lamp housing and take off the lamp cover.
- Twist the lamp 90° and slide it straight out.
- Insert the new lamp and rotate it 90° in the reverse direction to secure it in place. Make sure that the lettering on the lamp will be facing to the left of the instrument when it has been reinstalled. If it is not, remove the lamp and reinsert it correctly.
- Replace the optical assembly.

7.5.5 Cleaning the Optical Cell

If the relative absorbance of standards differs significantly from that of previous calibrations, the optical cell (located inside the front panel) may be dirty and must be cleaned:

- Turn the lamp and the power off and remove the front panel by lifting it up and out.
- Remove the optics clamps, disconnect the detector, and rotate and lift out the assembly. Disconnect the gas lines.
- Remove the six screws holding the lamp spacer and the detector spacer onto the optical cell.

- Inspect the two ends with the lenses. If the external surface of the lenses appear to be the only contaminant, then clean. To clean use methanol. Install if no other cleaning is necessary.
- Disassemble the optical cell (using the allen wrench provided on the inside of the front cover) by removing (in order) the screws, lens, and gasket at each end.
- Carefully clean the inside of the cell with laboratory glassware cleaner, taking care not to scratch the inside surfaces. Rinse thoroughly, first with water and then with DI water. Dry the cell in the oven (free of contaminants) for one hour at approximately 40 - 50°C.
- Clean the lenses with laboratory glassware cleaner and rinse thoroughly with hot tap water. Flush lightly with methanol and dry by air or vacuum oven (maximum 50°C).
- Replace the gaskets (this is recommended although not required unless the gasket shows signs of wear) and reassemble the optical cell. Cleaning of the gaskets should only be done with DI water.

7.5.6 Replacing the Liquid Gas Separator

- The liquid gas separator (transparent block on the chemical panel) should only need to be replaced once every one to three years, depending on the amount of use it receives.
- To replace the separator, shut off the gas and liquid flow and flush the tubing with DI Water for safety purposes. Disconnect the four lines and remove the two screws. Remove the unit from the system, screw on a new one, reconnect the four lines, and turn the gas and liquid flow back on.

7.5.7 Replacing Internal Tubing

Internal gas and Teflon tubes should last indefinitely and should not need to be replaced. Periodically inspect all tubing for restrictions or blockages. If tubing should need to be replaced, do so one piece at time to avoid any confusion while making connections.

7.6 Sample Analysis

7.6.1 Preparing the System

The following procedures must be performed each morning before warming up the system:

- Press the F10 macro key to stop any currently running macro.
- Change the drying tube. Refer to maintenance, Section 7.5 for instructions.
- Release the clamps and check the pump tubing for wear. Under normal use, the tubes will need to be replaced once a week. To replace the tubing, refer to maintenance, Section 7.5 for instructions.
- Check the reductant volume and refresh, if needed.
- Clean the rinse tank using standard lab cleaning practices, add fresh rinse.

- If the lamp has been off then turn on the lamp power and allow the lamp to warm up for at least 45 minutes.
- If the system is shut off, power up all components and perform COLDSTRT macro.
- Start up the system.

7.6.2 Start-up Procedures

The start-up routine used will depend on the current state of the system. If it is in Overnite mode, use the Warmstart macro (Section 7.6.3). If the system has been completely powered down, run the Coldstart macro instead (Section 7.6.4).

7.6.3 Warm Start

- The Warmstart macro is used to prepare the instrument for operation if it is being started up from a short-term (overnight) shutdown.
- To run the Warmstart macro, press the F2 macro key on the keyboard. Type WARMSTRT and press ENTER. The system will wait for several minutes and then turn on the pump and the gas flow to protocol speed. When the system is stable, a beep will sound and an "Operation Complete" message will appear on the screen. The instrument is now ready for operation.

7.6.4 Cold Start

- The Coldstart macro procedure is used to prepare the instrument for operation if the system has been shut down for an extended period of time. This procedure turns on the liquid and the gas flow and then waits until the system thermally equilibrates before beeping to indicate that it is ready to run. Perform an aperture test and make any necessary adjustments to the aperture before analyzing samples.
- To run the Coldstart macro, press the F2 macro key on the keyboard, type COLDSTRT and press ENTER. The Coldstart procedure takes approximately 2 1/2 hours. Do not attempt to operate the instrument before this procedure is complete, or its performance will be significantly impaired.
- When a beep has sounded and an "Operation Complete" message is visible on the screen, indicating the completion of the Coldstart procedure, check the apertures on the optical cell and make any necessary adjustments; this procedure is documented in Section 2.10, steps 1 and 2 of the operator's manual. When the aperture adjustments are completed, the instrument is ready for operation.

7.6.5 Software Setup

- In order to run samples, enter all necessary information regarding the protocol, sample ID's, calibration values, and autosampler parameters into the software. This information is entered into a series of screens which are accessed from the Main Menu. (Display the Main Menu at any time by pressing the F1 key)
- Perform each of the following steps in sequence to set up the software. When these steps have been completed, the instrument will be able to run samples automatically.

NOTE: The steps below comprise the basic daily software setup sequence. The software also contains numerous advanced functions. Refer to the PS Series Reference Guide for a detailed description of the many other keys and functions available for use with this system.

7.6.6 Name the Protocol

Protocols are operational determinations (parameters) for running calibrations and samples. Name the desired protocol to instruct the instrument what its normal operational values will be for running the next batch of samples.

- From the Main Menu, select PROTOCOL and then select GET. The Protocol screen will appear a "Get protocol name:" message will be displayed at the bottom of the screen.
- Type the protocol name and press ENTER. This creates a protocol file.
- Press the F1 key to return to the Main Menu.

7.6.7 Name the Folder

Once the protocol has been named, create a folder to hold all data generated from each sequence of operation.

- From the Main Menu, select DATA OUTPUT and then select Open folder. The Folder maintenance screen appears and an "Enter folder name:" message will be displayed at the bottom of the screen.
- Type a folder name and press ENTER. The folder is created.
- Press the F1 key to return to the Main Menu.

7.6.8 Verify Values and Integration Times

Check to make sure that all values and integration times are correct for running the samples:

- From the Main Menu, select PROTOCOL, then select SET Values. The Set Values screen appears.
- For normal operation, enter the following values (as Illustrated below):

Number of Integrations:	1
Uptake time	10
Weight	N
Dilution	N
Percent Recovery	N

- Press F1 to return to the Main Menu.

7.6.9 Enter values for on/offs, times, and gains

- From the Main Menu, select PROTOCOL, then select ON/OFFS, TIMES, GAINS. The on/offs, times, gains screen appears and an "Enter integration time:" message is displayed at the bottom of the screen.
- Type the desired integration time from between 1 and 30 seconds (the typically selected value is 10 seconds) and press ENTER.
- Press the F1 key to return to the Main Menu.

7.6.10 Enter the Calibration Standard Concentrations:

- From the Main Menu, in sequence, select CALIBRATION, STANDARDS, and then UNITS. The Units screen appears an "Enter units:" prompt is displayed at the bottom of the screen.
- Type the desired unit of measurement (e.g., ppb) and press ENTER. The entry will appear in the Units column above.
- Using the hot key, select each standard on the screen (S1-S6) and enter the appropriate calibration standard concentration (e.g., S1-.00000, S2-.20000, S3-.50000, S4-1.0000, S5-3.0000, S6-5.0000)
- Press the F1 key to return to the Main Menu.

NOTE: Do not be concerned with the UI (Update Intercept) and US (Update Slope) columns at this time. If more information is required in these fields, refer to the PS Series Reference Guide.

7.6.11 Reset the Calibration Intensity Data

- From the Main Menu, select CALIBRATION, RESET, and NEW CALIBRATION RESET. The Reset screen appears at the bottom of the screen.
- To erase any calibration data that may have already been done with this protocol, enter Y and press ENTER. An "All Data Reset" message will appear when the process is complete. (To escape this procedure, enter N instead.)
- Press the F1 key to return to the Main Menu.

7.6.12 Set the Autosampler Rinse Time

- From the Main Menu, select AUTOSAMPLER, SETUP, and RINSE TIME (seconds). The Setup screen appears and an "Enter rinse time:" message is displayed at the bottom of the screen.
- Type the desired value in seconds (typically 50) and press ENTER.
- Press the F1 key to return to the Main Menu.

7.6.13 Set up the Racks

- From the Main Menu, select AUTOSAMPLER and then RACK ENTRY. The Rack screen appears and an "Enter rack name:" message is displayed at the bottom of the screen.
- Type a rack name (either new or existing) and press ENTER. (If a new name is entered, a prompt will appear to ask if you want to create a new rack: answer Y.)

- Fill the sample cups to be used to within 1/4" from the top (to allow for two runs). Using the autosampler layout in as a guide, load each sample cup into the rack and enter the sample ID into the appropriate (cup) position on the rack entry screen.

NOTE: For details on the INSERT key, rack calculation options, and advanced editing options, refer to the PS Series Reference Guide.

- It is important to remember that the instrument can run two complete racks unattended.
- Press the F1 key to return to the Main Menu.

7.6.14 Define start-to finish sample sequence

- From the Main Menu, select AUTOSAMPLER and then SETUP. Type the rack number to be run (1 or 2). The prompt "Enter rack name" is displayed at the bottom of the screen.
- Type the rack name and press ENTER. The Setup screen for that rack will appear and a "Begin cup:" prompt will be displayed at the bottom of the screen.
- Enter the number (cup position) of the first cup to be sampled and press ENTER. An "End cup:" prompt will now be displayed at the bottom of the screen.
- Enter the number of the last cup to be sampled and press ENTER.
- Press the F1 key to return to the Main Menu.
- If using a second rack, repeat steps 1-5.

7.6.15 Running Samples

NOTE: Optimum Concentration Range = 0.2 ug/L - 5 ug/L

- Press the F8 macro key. The Autosamples setup menu appears and a "Press F8 again to run sample" message will be displayed at the bottom of the screen.
- Press the F8 A Macro key again. The instrument will run the samples, print the results, and store the data in the folder that was created.
- When all samples have been run, the system will beep and the word "Idle" will appear in the State field at the top of the screen. At this time, repeat the above steps to run more samples or shut down the instrument. Refer to Section 7.6.16 for shutdown procedures.

NOTE: Each sample takes ~2 minutes to run: a full tray (88 samples) will take ~2 1/2 hours to complete. As operation is fully automatic, laboratory personnel need not be present while samples are running.

7.6.16 Shutdown Procedures

There are two methods for shutting down the instrument. Under routine operation, when the system is used daily, only the lamp is shut off (system power remains on) and the

Overnite routine is used to put the unit into a "sleep mode". If the system is to be completely turned off and not used for an extended period of time , or if it is to be shipped or moved, use the long-term Shutdown routine instead. These two methods are described below. For weekends or periods of "sleep" greater than 24 hours it is recommended to turn off the mercury lamp using the blue button.

NOTE: Before shutting down the instrument, the system must have beeped to indicate completion of the last procedure, and the word "Idle" should appear in the "State field in the top left of the displayed screen.

7.6.17 Short-Term (Overnite Macro)

Press the F2 macro key, type OVERNITE, and press ENTER. Turn off power to the lamp if the instrument will not be used for longer than 24 hours. In overnite mode, the pump and gas flow will turn on every few minutes, run for a few seconds and then stop. This cycle exercises the tubes so they don't get flat spots and fatigue, and the gas flow keeps the optical cell dry.

SUGGESTION: If macros are used to automate the run procedures, call the Overnite procedure at the end (CM....) so that the system will shut down automatically when the last procedure is finished.

7.6.18 Long-Term (Shutdown Macro)

The Shutdown macro procedure is designed to flush out all lines with DI water to get rid of any chemical residues.

- Lift the sample tip and remove the rinse tray. Rinse and fill it with DI water and replace the tray. Lower the sample tip into the cleaned tray.
- Remove the reductant bottle cap and line and carefully place the tip of the line in the rinse tank (rest the cap on the corner of the tray).
- Turn off the lamp.
- Press the F2 macro button. Type SHUTDOWN and press ENTER. When a "beep" is heard and the word "Idle" appears in the State field at the top left of the screen (wait several minutes), release all pump clamps.
- Remove the front cover of the instrument and remove the optical cell (refer to Section 7.5). Disconnect the two gas lines on the left side of the cell and leave them hanging. Replace the optical cell and the front cover.

NOTE: The next time the system is started up, remember to re-open the front cover, remove the optical cell and reconnect the gas lines.

- Shut off power to the computer, monitor, printer, and finally the instrument itself.

7.7 Documentation

7.7.1 Instrument Run-Log

The analysis of samples and standards is documented within the instrument run log (Attachment 1) and supported by the instrument print-out. The runlog must be completed for each days analysis.

7.7.2 Traceability of Standards

Custom made and single element stock standard solution which are traceable to NIST or EPA are purchased. On receipt, each standard is recorded in LabNet (LIMS) and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, date of verification and the initials of the recording analyst are entered into the system.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. The analyst and a trained data reviewer perform the reviews according to the criteria established on the data review checklist (Attachment 2). Upon the first 100% review, the checklist is initialed and dated as reviewed. The package, with its review sheet, comments and any corrective action reports (CARs) is submitted to the supervisor, section manager, or peer reviewer for a second review. Once again, the checklist is initialed and dated by the second reviewer. The completed checklist remains on file with the original data.

8.0 QUALITY CONTROL

8.1 QC Summary

The laboratory generates annual statistically generated control limits and these can be used when requested by the client, contract or QAPP. These limits are based on the successive analysis of LCSs.

8.1.1 Calibration curve must be composed of a minimum of a blank and 5-standards. A least square fit linear calibration curve must have a minimum correlation coefficient of 0.995, which must be reported with the raw data.

8.1.2 ICV and ICB will be performed at the beginning of an analytical sequence. The ICV must not vary more than a) 10% for SW-846 & CLP methods or b) 5% for EPA Methods from its true value and must be prepared from a different source than the calibration curve standards.

Calibration verification will be performed with a CCV and CCB every 10 samples and at the end of the analysis. The CCV must not vary more than a) 20% for SW-846 methods or b) 10% for EPA & CLP methods from its true value and must be prepared from a different source than the calibration curve standards. The CCB must be < Reporting Limit (EPA / SW-846) / CRDL (CLP).

8.1.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve (dilute with a digested blank containing all reagents, or repeat the analysis using a smaller sample volume).

8.1.4 A minimum of one MB must be analyzed per sample batch to determine if contamination has occurred

8.1.5 An LCS will be included with each batch of 10 (drinking waters and EPA 245.1) or 20 (SW-846 or CLP) samples. The analyzed result must not vary more than 20% from the true value. For EPA Method 245.1, the LCS acceptance limits are 85-115%.

8.1.6 Matrix spike and duplicate samples are analyzed with each batch of 10 (drinking waters and EPA 245.1) or 20 (SW-846 or CLP) samples.

8.2 Corrective Actions

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out of control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, section manager, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out of control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the supervisor, section manager, QA personnel or the laboratory manager.

Listed below are steps that must be taken when an out of control situation occurs:

- demonstrate that all the problems creating the out of control situation were addressed
- document the problem and the action which was taken to correct the problem on a CAR
- document on the CAR that an in control has been achieved and receive approval (signature) of the supervisor, section manager, QA personnel, or the laboratory manager prior to the release of any analytical data associated with the problem.

8.2.1 Calibration Curve

- reanalyze the standard curve;
- prepare new stock and/or working standards;
- check reagents/solutions and prepare fresh if necessary.

8.2.2 Initial Calibration Verification (ICV)

- repeat ICV to verify proper preparation;
- prepare new ICV from original stock;
- recalibrate with a new standard curve;
- prepare new stock and/or working standards;
- check reagents/solutions and prepare fresh if necessary.

8.2.3 Initial Calibration Blank (ICB)

- prepare new ICB to verify proper preparation;
- verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability;
- determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem;
- check reagents/solutions and prepare fresh if necessary;
- correct for any contamination and reanalyze ICB and any associated samples.

8.2.4 Laboratory Control Sample (LCS)

If LCS is low:

- reanalyze LCS to verify that it is out of control;
- determine the source of error within the preparation procedure, repeat the sample set, write a CAR.

If the LCS is high:

- reanalyze LCS to verify that it is out of control;
- determine the source of error within the preparation procedure, repeat the sample set;
- determine if the high result is due to contamination;
- check for contamination of reagents, LCS stock solution, or preparation area;
- correct for contamination, reanalyze.

8.2.5 Method Blank (MB)

- reanalyze MB to verify that it is beyond the reporting limit;
- determine the source of contamination;
- determine if the high result is due to contamination;
- check for contamination of reagents or preparation area;
- correct for contamination, reanalyze set;
- in the extreme case where all samples in the set are at least 10X > the MB, reanalysis will not be required. However, a CAR and approval will be necessary.

8.2.6 Matrix Duplicate (MD)

- the sample must be reprocessed and reanalyzed;
- if the reanalysis results in data that is still out of the control limit, then the sample will be ticked with a "*";

- regardless of the outcome of the reanalysis, a CAR will be written and approved by the Section Manager.

8.2.7 Matrix Spike (MS)

- the sample must be reprocessed and reanalyzed;
- if the reanalysis results in data that is still out of the control limit, then the sample will be ticked with a "N";
- regardless of the outcome of the reanalysis, a CAR will be written and approved by the Section Manager.

8.2.8 Continuing Calibration Verification (CCV)

- repeat CCV to verify proper preparation;
- prepare new CCV from original stock;
- check for instrument base-line drift or a change in one or more of the reagents;
- check reagents/solutions and prepare fresh if necessary;
- recalibrate with a new standard curve and repeat all samples since the previous in control CCV;
- never dispose of any samples until you are sure that all QC, especially the CCV, are within the control limits.

8.2.9 Continuing Calibration Blank (CCB)

- prepare new CCB to verify proper preparation;
- verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc.. to achieve stability;
- determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem,
- check reagents/solutions and prepare fresh if necessary;
- correct for any contamination and reanalyze CCB and any associated samples;
- never dispose of any samples until you are sure that all QC, especially the CCB are within the control limits.

8.2.10 Summary

- If any of the ICV, ICB, CCV or CCB results are out of control for any element, the instrument is restandardized and the samples associated with the out of control elements are reanalyzed.
- If the MB or LCS are out of control for any element, the samples are redigested. An exception is if the sample concentrations are $\geq$ 10X the MB contamination, the results are reported as is.
- If any of the MD or MS results are out of control, a reanalysis is performed if there is sufficient sample. If there is insufficient sample, or the reanalysis is still out of control, the client is notified of the poor results via a case narrative that is sent with the data report.
- CARs are available for out-of-control MB, LCS, MS and MD problems. These forms are completed by the analyst performing the analysis. The forms are then reviewed

and signed by the supervisor or section manager. The signed forms are kept on file within the laboratory department and are used to prepare the case narrative (if applicable).

9.0 DATA ANALYSIS AND CALCULATIONS

Perform a linear regression or quadratic fit analysis of the calibration standard results. Compare sample results to the curve to determine the mercury concentration.

9.1 Water ug/L Hg = ug/L x Dilution Factor (Where L = Final digestate volume)

9.2 Soil $$\text{mg/kg Hg} = \frac{(\text{ug/L}) \times \text{L} \times \text{Dilution Factor}}{\text{wt(g)} \times \text{fraction solids}}$$

(Where L = Final digestate volume)

NOTE: All dry weight corrections are made in LabNet at the time the final report is prepared.

9.3 Accuracy $$\%R = \frac{(A_T - A_O)}{A_F} \times 100$$

Where:
A_T = Total amount recovered in fortified sample
A_O = Amount recovered in unfortified sample
A_F = Amount added to sample

9.4 Precision $$RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$$

Where:
C_1 = First measurement value
C_2 = Second measurement value

10.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- Waste from this process goes into the " Corrosive Wastewater" wastestream.
- Single component standards should not be mixed into the waste streams unless approved by the Waste Coordinator. All standards with Hazardous constituents will be turned in to the waste technician for disposal.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Attachment 1: Example: Analysis Instrument Runlog
Attachment 2: Example: Data Review Checklist

Historical File:

Revision 00: 10/03/90
Revision 01: 08/09/91
Revision 02: 03/19/93
Revision 03: 10/18/95
Revision 04: 01/24/97
Revision 05: 03/31/99
Revision 06: 03/16/00
Revision 07: 05/23/01
Revision 08: 09/06/02

Reasons for Change, Revision 08:

- Updated the Health & Safety (3.0) and Waste Disposal (10.0) sections.
- Section 7.1: Amended statement regarding MS/MSD and MS/MD.

U:\QC\SOP\ME\2451.DOC

Attachment 1:

Example: Analysis Runlog

STL Chicago
Mercury Digestion Log / Analytical Run Log

Page No.:______________
Book #:______________
LabNet File:______________

Circle Method:
a: Water: EPA 245.1 / SW-846 7470A
b: Soil/Solid: EPA 245.5 / SW-846 7471A
c: Water/Soil/Solid: U.S. EPA SOW ILM04.0
d. Other: ______________________

Instrument: Leeman Labs PS200 (HG4)
Wavelength: 253.7 nm
Optical Cell Length: 20.5 cm

Standard/Matrix Spike Source ID#:__________ ICV/CCV/LCS Source ID#:__________ Equip. ID#:________
Water Bath Temp.: Initial:_______°C Final:_______°C Control Limits: 90°C to 95°C H_2SO_4 Lot #:_________
Thermometer ID:________ Correction Factor:_______°C HNO_3 Lot #:_________ HCl Lot #:__________

Repipettor Volume Check:
☐ HNO_3, 2.5 mL ☐ H_2SO_4, 5.0 mL ☐ $KMnO_4$, 15 mL ☐ $K_2S_2O_8$, 4.0 mL ☐ $NH_2OH \cdot HCl$, 6.0 mL

Comments:____

AS Pos. #	Sample # / QC ID	Sample Size (mls or g per final vol [1])	% Solids	Anal. Dil.	Comments

QC2 ICV/CCV:______________ 100 / 25 mLs ______________
QC1 ICB/CCB:______________ 100 / 25 mLs ______________

QC2 CCV:______________ 100 / 25 mLs ______________
QC1 CCB:______________ 100 / 25 mLs ______________

[1] Water bath digestion requires 100 mL final volume; Hot block digestion requires a 25 mL final volume.

Preparation Signature:______________________ Date:________ Time In:______ Time Out:_____

Analyst Signature:______________________ Date Analyzed:________ Date LIMS'd:________

Reviewer Signature:______________________ Date:______________

Attachment 2.

Example: Data Review Checklist

STL Chicago
Mercury Data Review Checklist: Automated CV (PS 200)

Instrument ID: HG3 HG4 (circle one) **LabNet Batch No.:**__________________

Analyst Initial(s):________________________ **Date:** __________________________

Copies: ________________________ __________________________

****

QC Type: a. CLP b. Standard c. TCLP d. Drinking Waters e. Solubles

I. Calibration:
Analyst Reviewer

1. Calibration is clearly documented.
   - ___ ___ a. c.c.: 0.995 to 1.000
   - ___ ___ b. y-intercept: **Std. QC**: < RL; **CLP QC**: < CRDL

2. Calibration Verification
   CLP QC: Every 10 Sample Bottles
   Std. QC: Every 20 Sample Bottles
   TCLP QC: Every 20 Sample Bottles
   Drinking Waters: Every 10 Sample Bottles
   - a. ICV/CCV **Std./SW-846:** ± 10% (ICV); ± 20% (CCV);
     CLP: ± 10% (ICV); ± 10% (CCV)
     EPA 245.1/245.5: ± 5% (ICV); ± 10% (CCV)
   - ___ ___ b. ICB/CCB **Std. QC**: < RL; **CLP QC**: < CRDL

3. CRA
   ___ ___ **CLP QC**: At CRDL; Analyzed each Calibration; No Limit; **Std. QC**: At CRDL; Analyzed Daily; ± RL

II. Sample Analysis:

Analyst Reviewer

1. Each Preparation Batch:
   - ___ ___ a. Must be clearly identified
   - ___ ___ b. Contains a maximum of 20 samples
   - ___ ___ c. 1 Prep Blank: **CLP:** < CRDL; **Std. QC:** < RL
   - ___ ___ d. 1 LCS: **Std./CLP:** 80-120% Rec. **EPA 245.1:** 85-115% Rec.
   - ___ ___ e. 1 Matrix Spike: **Std./CLP:** 75-125% Rec.; ***Unless*** the sample conc. exceeds the spike conc. by factor 4x. **TCLP:** > 50% Rec.; If <50%, MSA analysis is required
     245.1: 85-115% Rec.
   - ___ ___ f. 1 Matrix Duplicate: **Std.:** RPD/RSD limits are 20% ***Unless*** the sample conc. is <5x RL then ± RL
     CLP: RPD or RSD limits are 20%; ***Unless*** the sample conc. is <5x CRDL then ± CRDL applies.
   - ___ ___ g. % TS for samples to be reported on a Dry Wt.

III. Data Documentation
Analyst Reviewer

1. ___ ___ The instrument and current conditions must be clearly documented. The Temperature of the Water Bath must be 95°C.
2. ___ ___ **All** Percent Recoveries and RPD's need to be documented in the raw data.
3. ___ ___ If the CCB/PB and/or CCV/LCS are outside of the control limits, **a CAR must be written** and the Section Manager or Unit Leader must be notified that redigestion is required.

III. Data Documentation (continued)

Analyst Reviewer

4. Matrix Spike outside the control limits:
   a. **CLP QC**: No corrective action required, the sample is ticked appropriately.
   b. **Std. QC**: **A CAR must be written** and the Section Manager or Unit Leader must make the decision as to whether re-digestion is required.
   c. If MSA is performed; check the calculation.
5. Sample Duplicate outside the control limits:
   ___ ___ a. **CLP QC**: Normally no corrective action required, and the result is ticked appropriately.
   ___ ___ b. **Std. QC**: **A CAR must be written** and the Section Manager or Unit Leader must make the decision as to whether redigestion is required.

___ ___ 6. The sample data and QC is recorded in the databook in the order in which they were analyzed. **All** unused data is clearly identified.

___ ___ 7. Standard Traceability is correctly documented.

___ ___ 8. Data Report accurately reflects the documentation in the Databook and the LIMS Spreadsheet.

9. The analyst's full signature is required on the following:
   ___ ___ a. Instrument Data Report
   ___ ___ b. Databook
   ___ ___ c. Data Review Checklist
   ___ ___ d. Print out LabNet Pages, Raw Data, QC, and RunLog
   ___ ___ e. Samples needing copying are clearly marked

___ ___ 10. All unused portions of the data page are Z'd out.

___ ___ 11. Proper Corrective Action Documentation for any out of control situation is clearly identified.

IV. Miscellaneous

Analyst Reviewer

___ ___ 1. Is Sample Prep Linked?

___ ___ 2. Is TCLP Linked? (Shift F9 from the start page)

___ ___ 3. *Did all dilutions carry over for MD, MS, MSD (where applicable)?*

___ ___ 4. Did all prep and analysis matrices match up?

Comments:____

Analyst Signature: ______________________________ Date:

Reviewer Signature: ______________________________ Date:

STL CHICAGO
LABORATORY STANDARD OPERATING PROCEDURE

SOP No.	Revision No.	Date	Page
USP-1311	07	06/01/01	1 of 20

TITLE: SAMPLE PREPARATION
Toxicity Characteristic Leaching Procedure (TCLP)

Updated by:	Signature:	Date:
Paul Mattei TCLP Analyst	Paul Mattei	5/31/01

Approved by:	Signature:	Date:
Mani S. Iyer Section Manager, Metals Dept.	Mani S. Iyer	5/31/01
Raymond J. Frederici Quality Manager	Raymond J. Frederici	5-31-01

CONTROLLED DISTRIBUTION

COPY # : **Uncontrolled**

ISSUED TO : **Mark Densmore, Secor**
Re: UTC Proposal

Full Signature Approvals Are Kept on File
with STLs QA Standard Practice Records

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for the Toxicity Characteristic Leaching Procedure (TCLP). This SOP was written using 40 CFR 261 (Appendix II) and SW-846, 3rd Edition, Method 1311 as reference.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable. Refer to the analytical SOPs.

1.1.2 Reporting Limits

Not Applicable. Refer to the analytical SOPs.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM, Revision 01).

1.2 Summary of Method

TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solids and multi-phasic wastes.

Two distinct methods are utilized depending on whether volatile organics or other organic and metal constituents will be analyzed. A special zero-headspace extractor (ZHE) is used for volatile sample preparation and 2.0-Liter HDPE plastic or Teflon bottles are used for the other constituents.

- For solid wastes or wastes that contain significant amounts of solid material, the particle size is reduced and the liquid phase (if any) is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid that is equal to 20 times the weight of the solid material.

- For VOA's, the liquid and solid phases are separated by filtering prior to and after the extraction. For all other parameters, the liquid and solids phases are separated after the extraction

- A portion of the extract for metals analysis <u>only</u> are spiked by the TCLP analyst with the analytes of concern (at the regulatory level) and acidified with nitric acid to a pH < 2. (Refer to Appendix A for this procedure.)

The TCLP sample is then analyzed by the appropriate method for organic and metal constituents. Refer to Figure 1 for the TCLP Flowchart; Table 1 for a listing of the Toxicity Characteristic Constituents and Regulatory Levels; and Table 2 for the maximum sample holding times.

2.0 INTERFERENCES

- Since this is a preparation procedure, interferences will only become apparent at the spiking and analysis stage. Interferences for spiking and for instrumentation are discussed in the analytical SOPs.
- A physical interference may occur for pH readings if the waste material is high in organic material (such as an oil). The waste may coat the pH probe, which affects the ability to obtain an accurate reading. When this type of interference occurs, pH paper is used instead of a meter for the final pH measurement. The use of pH paper is documented in the laboratory logbook.

3.0 SAFETY

- As always, general laboratory safety practices should always be followed. Waste samples should be handled with care due to the uncertainty of the properties and contents involved.
- Refer to the specific Material Safety Data Sheets (MSDSs) for the hazardous properties of any chemical or reagent involved in this procedure.
- Acids should be handled with care.
- Since all samples that are being tested may contain hazardous substances, care should be taken to avoid contact with the samples or the filtrates.

4.0 EQUIPMENT AND SUPPLIES

- The extractor is a custom made rotary type design that meets the specifications of tumbling the samples at a rate of 30 ± 2 RPMs.
- 2-Liter Nalgene bottles (HDPE for metals)
- 2-liter Teflon bottles [For organics (BNA, Herb/Pest)]
- pH meter and paper - pH meter accurate to ±0.05 pH units at 25°C. Refer to the pH SOP (UWC-SOP-150.1) for details on meter calibration.
- Filtering apparatus - pressure filter using compressed Nitrogen as the purge gas
- Zero Headspace extraction vessel (ZHE) - purchased unit for volatiles
- Filter paper - glass fiber, 0.7 um pore size.

NOTE: Filters shall be made of borosilicate glass fiber. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1 N nitric acid followed by 3 consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended).

- Lab balance capable of reading ± 0.01 g
- *Tedlar Bags *Registered Trademark
- ZHE Extraction Fluid Transfer Device - any device capable of transferring the extraction fluid to the ZHE without changing the nature of the extraction fluid is

acceptable (e.g., a positive displacement or a peristaltic pump, a gas tight syringe, pressure filtration unit).

5.0 REAGENTS AND STANDARDS

5.1 Hydrochloric Acid, 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, carefully add 83 mLs of concentrated hydrochloric acid. Swirl the flask to mix. Dilute to volume with Milli-Q water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.2 Nitric Acid, 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, carefully add 64 mLs of concentrated nitric acid. Swirl the flask to mix. Dilute to volume with Milli-Q water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.3 Sodium Hydroxide, 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, add 40.0 g of sodium hydroxide pellets. Swirl the flask to mix. This is an **EXOTHERMIC** reaction. The flask should be placed in a cool water bath when mixing. Dilute to volume with Milli-Q water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.4 Glacial Acetic Acid, Reagent Grade

Purchased.

- Life of Reagent: 1 year
- Storage Requirements: None

5.5 Extraction Fluid #1

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, carefully add 5.7 mLs of glacial acetic acid. Swirl the flask to mix. Then add 64.3 mLs of 1.0 N sodium hydroxide solution (Rgt. 5.3) and swirl to mix once again. Dilute to volume with Milli-Q water. The pH of this extraction fluid should be 4.93 $\pm$ 0.05.

- Life of Reagent: 1 day
- Storage Requirements: None

5.6 Extraction Fluid #2

To a 1-L Class A volumetric flask containing ~500 mLs of Milli-Q water, carefully add 5.7 mL of glacial acetic acid. Swirl the flask to mix. Dilute to volume with Milli-Q water. The pH of this Extraction Fluid should be 2.88 $\pm$ 0.05.

- Life of Reagent: 1 day
- Storage Requirements: None

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

Refer to Section 8.1.

7.2 Sample Preservation and Storage

Parameter	From: Field Collection To: TCLP Extraction	From: TCLP Extraction To: Preparative Extraction	From: Preparative Extraction To: Determinative Analysis	Total Elapsed Time
Volatiles	14 days	NA	14 days	28 days
Semi-Volatiles	14 days	7 days	40 days	61 days
Mercury	28 days	NA	28 days	56 days
Metals (except Hg)	180 days	NA	180 days	360 days

NA = Not Applicable

7.3 Sample Preparation / Size

7.3.1 Inorganics & Semi-Volatiles

Type of Sample	Sample Size
Samples containing 100% solids	100g solid
Samples containing 0.5% - 99.9% solids	100 g solid ideally, 75.0 g solid minimum

7.3.2 Organics & Volatiles

Type of Sample	Sample Size
Samples containing 100% solids	25 g solid
Samples containing 5% - 99.9% solids	25 g solid
Samples containing <5% solids	500 g solid

7.4 Calibration / Standardization

Refer to SOP No. UWC-150.1 for instructions on calibrating the pH meter.

7.5 Preventive Maintenance

- The main preventive maintenance required is keeping the area and all equipment clean and free of contaminants.
- The pH probe should be checked periodically for bubbles. The probes are replaced when needed.
- The ZHE's shall be checked for leaks after every use.

7.6 Sample Extraction

7.6.1 Procedure when Volatiles are Not Involved

Although a minimum sample size of 100 grams is required, a larger sample size may be necessary, depending on the percent solids of the waste sample. Enough waste sample should be collected such that at least 75 grams of the solid phase of the waste (as determined using glass fiber filter filtration) is extracted. This will ensure that there is adequate extract for the required analyses (semivolatiles, metals, pesticides and herbicides).

The determination of which extraction fluid to use (sec. 7.6.1.12) may also be conducted at the start of this procedure. This determination shall be on the solid phase of the waste (as obtained using glass fiber filter filtration).

7.6.1.1 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative 100.0 g portion of the sample and proceed to 7.6.1.11.

7.6.1.2 If the sample is liquid or multi-phasic, liquid/solid separation is required. This involves the filtration device outlined in secs. 7.6.1.3 through 7.6.1.9.

7.6.1.3 Pre-weigh the filter and the container which will receive the filtrate.

7.6.1.4 Assemble the filter holder and filter.

7.6.1.5 Weigh out a representative 100 g sub-sample of the waste and record the weight.

7.6.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.6.1.7 Transfer the waste sample to the filter holder.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in sec. 7.6.1.5 to determine the weight of the waste sample which will be filtered.

Gradually apply pressure of 10 psi, until gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any two minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter in any two minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi, filtration is stopped.

7.6.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes will obviously contain some material that appears to be a liquid - but even after applying pressure filtration this material may not filter. In this case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid.

7.6.1.9 Determine the weight of the liquid phase by subtracting the total weight of the filtrate container (sec. 7.6.1.3) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (sec. 7.6.1.15) or stored at 4 ± 2°C until time of analysis.

The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in sec. 7.6.1.5 or 7.6.1.7. Record the weight of the liquid and solid phases.

NOTE: If the weight of the solid phase of the waste is < 75 g. Review the beginning of section 7.3 about sample sizes.

7.6.1.10 The sample will be handled differently from this point, depending on whether it contains more or less than 0.5% solids. If the sample obviously has >0.5% solids, go to sec. 7.6.1.11. If it appears that the solid may comprise less than 0.5% of the total waste, the percent solids will be determined as follows:

- Remove the solid phase and filter from the filtration apparatus.
- Dry the filter and solid phase at 100 ± 20°C until two successive weighings yield the same value. Record the final weight.
- Calculate the percent solids as follows:

$$\frac{\text{(weight of waste \& filters) - (tared weight of filters)}}{\text{initial weight of waste}} \times 100 = \text{\% solids}$$

- If the solid phase comprises <0.5% of the waste, it is discarded and the liquid phase is defined as the TCLP extract. Proceed to sec. 7.6.1.14.
- If the solid is ≥0.5% of the waste, return to sec. 7.6.1.1 and begin the procedure with a new sample of waste. Do not extract the solid that has been dried.

7.6.1.11 If the sample has more than 0.5% solids, it is now evaluated for particle size. If the solid material is capable of passing through a 9.5 mm sieve, proceed to sec. 7.6.1.12. If the particle size is larger than 9.5 mm, the solid material is prepared for extraction by crushing until it is < 9.5 mm.

7.6.1.12 This step describes the determination of the appropriate extracting fluid to use.

- Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer a 5.0 g portion to a 250 mL beaker.

- Add 96.5 mL DI water, cover with watch glass, and stir vigorously for five minutes using a magnetic stirrer. Measure and record the pH. If the pH is $\leq$ 5.0, extraction fluid # 1 is used. Proceed to sec. 7.6.1.13.
- If the pH is >5.0, add 3.5 mL 1.0 N hydrochloric acid, stir for 30 seconds and heat to 50°C. Continue heating at 50°C for ten minutes.
- Let the solution cool to room temperature and record the pH. If pH is $\leq$ 5.0, use extraction fluid #1. If the pH is > 5.0, use extraction fluid #2.

7.6.1.13 Transfer the solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase.

NOTE: If any of the solid phase remains adhered to the walls of the filter holder, or the container used to transfer the waste, its weight shall be determined, subtracted from the weight of the solid phase of the waste, as determined above, and this weight is used in calculating the amount of extraction fluid to add into the extractor bottle.

Slowly add an amount of the appropriate extraction fluid into the extractor bottle equal to 20 times the weight of the solid phase that has been placed into the extractor bottle. Close the extractor bottle tightly, and place in the rotary extractor and rotate for 18 $\pm$ 2 hours. The ambient room temperature shall be maintained at 23 $\pm$ 2°C during the extraction period.

7.6.1.14 Following the 18 hour extraction, the material in the extractor vessel is separated into its component liquid and solid phases by filtering through a new glass fiber filter as outlined in Sec. 7.6.1.7.

7.6.1.15 The TCLP extract is now prepared as follows:

- If the waste contained no initial liquid phase, the filtered liquid material obtained from Sec. 7.6.1.14 is defined as the TCLP extract. Proceed to Sec. 7.6.1.16.

- If compatible (e.g., will not form a precipitate or has multiple phases), the filtered liquid is combined with the initial liquid phase of the waste. This combined liquid is defined as the TCLP extract.

- If the initial liquid phase of the waste, as obtained from Sec. 7.6.1.9 is not compatible with the filtered liquid resulting from Sec. 7.6.1.14, the liquids are not combined. The liquids are collectively defined as the TCLP extract and are analyzed separately.

7.6.1.16 The TCLP extracts are prepared according to the procedures for the particular analysis (organics or metals) before being analyzed. Following the collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and reserve for analysis (metals only). Metals must be acidified with Nitric Acid to pH <2. Refrigerate the aliquots at 4 ± 2°C.

7.6.2 Procedure for Volatiles by ZHE

The ZHE device has approximately a 500 mL internal capacity. Although a minimum sample size of 100 grams is required in Section 7.6.1, the ZHE can only accommodate a maximum 100% solids sample of 25 grams. This is due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Sec. 7.6.2.4 provides the means by which to determine the approximate sample size for the ZHE device. Although the following procedure allows for particle size reduction during the procedure, this could result in the loss of volatile compounds. If possible, any particle size reduction (see Sec. 7.6.2.5) should be conducted on the sample as it is being taken. Particle size reduction should only be conducted during the procedure if there is no other choice.

In carrying out the following steps, do not allow the waste to be exposed to the atmosphere for any more time than is absolutely necessary.

7.6.2.1 Pre-weigh the (evacuated) container which will receive the filtrate, and set it aside.

7.6.2.2 Place the ZHE piston within the body of the ZHE (it may be helpful to first moisten the piston o-rings slightly with extraction fluid). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set it aside. Set liquid inlet/outlet flange (top flange) aside.

7.6.2.3 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative 25 g sample of the waste, record the weight, and proceed to Sec. 7.6.2.5.

7.6.2.4 This sec. provides the means by which to determine the approximate sample size for the ZHE device. If the waste is liquid or multi-phasic, follow the procedure outlined in Steps 7.6.1.2 to 7.6.1.9 (using the Section 7.6.1 filtration apparatus), and obtain the percent solids by dividing the weight of the solid phase of the waste by the original sample size used. If the waste obviously contain >0.5% solids, go to Sec. 7.6.2.4. If it appears that the solid may comprise <0.5% of the waste, see below.

- Determine the percent solids by using the procedure outlined in Sec. 7.6.1.10. If the waste contains <0.5% solids, weigh out a 100 g minimum sample, proceed to Sec. 7.6.2.7 and follow until the liquid phase of the waste is filtered using the ZHE device (Sec. 7.6.2.8). This liquid filtrate is defined as the TCLP extract and is analyzed directly. If the waste contains > 0.5% solids, repeat Sec. 7.6.2.4 using a new 100 g minimum sample, determine the percent solids, and proceed on.

- If the sample is <0.5% solids, weigh out 500 g of sample and record the weight (proceed to Sec. 7.6.2.5).
- If the sample is $\geq$ 0.5% solids, the maximum amount of sample the ZHE can accommodate is determined by dividing 25 grams by the percent solids obtained from Sec. 7.6.2.4. Weigh out a new representative sample of the determined size by the following calculation:

$$\text{weight of waste to change ZHE} = \frac{25}{\text{percent solids}} \times 100$$

7.6.2.5 After a representative sample of the waste has been weighed out and recorded, the sample is now evaluated for the particle size (see beginning of Procedure 7.6.2). If the solid material within the waste will obviously pass through a 9.5 mm sieve, proceed immediately to Sec. 7.6.2.6. If the particle size is larger than that described above, the solid material which does not meet the above criteria is separated from the liquid phase by sieving, and the solid is prepared for extraction by crushing until the particle size is < 9.5 mm.

NOTE: Wastes and appropriate equipment should be refrigerated, if possible, to 4 $\pm$ 2°C prior to particle size reduction. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the furthest extent possible.

When particle size has been appropriately altered, the solid is re-combined with the rest of the waste.

7.6.2.6 Waste slurries should not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly shall not be centrifuged prior to filtration. Again, this is to minimize the loss of volatile compounds to the atmosphere.

7.6.2.7 Transfer the entire sample (liquid and solid phases) quickly to the ZHE. If there is no solid/liquid separation, proceed to sec. 7.6.2.11.

Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Sec. 7.6.2.4, to determine the weight of the waste sample which will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange), and with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (more if necessary) to slowly force all headspace out of the ZHE device.
At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

7.6.2.8 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open valve. Begin applying gentle pressure of 1 - 10 psi to force the liquid phase into the filtrate collection container. If no additional liquid has passed through the filter in any two-minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi.

After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any two-minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within any two-minute period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.6.2.9 The material in the ZHE is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid - but even after applying pressure filtration this material will not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% solids (see Sec. 7.6.2.4) this filtrate is defined as the TCLP extract, and is analyzed directly - proceed to Sec. 7.6.2.13.

7.6.2.10 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Sec. 7.6.2.1) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed or stored at 4 $\pm$ 2°C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample (see Sec. 7.6.2.4). Record the final weight of the liquid and solid phases.

7.6.2.11 The following details how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel.

Extraction fluid #1 is used in all cases.

- With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be pre-flushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid into the ZHE. Continue pumping extraction fluid into the ZHE until the amount of fluid introduced into the device equals 20 times the weight of the solid phase of the waste that is in the ZHE.

- After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to make sure that all valves are in their closed positions. Pick up the ZHE and physically rotate the device in an end-over-end fashion two or three times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top.

  Put 5-10 psi behind the piston and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This is a check to show that the piston moves under 15 psi and that the o-rings are ok. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

- Place the ZHE in the rotary extractor apparatus and rotate the ZHE for 18 $\pm$ 2 hours. The temperature of the room shall be maintained at 23 $\pm$ 2°C during agitation.

7.6.2.12 Following the 18 hour extraction, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release is observed) the device is leaking. Replace ZHE o-rings or other fittings, as necessary, and re-do the extraction with a new sample of waste. The original extract can not be used. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container holding the initial liquid phase of the waste, unless doing so would create multiple phases, or unless there is not enough volume left within the filtrate collection container. A separate filtrate collection container must be used in these cases. Filter through the glass fiber filter, using the ZHE device as discussed in Sec. 7.6.2.8.

7.6.2.13 If the waste contained no initial liquid phase, the filtered liquid material obtained from Sec. 7.6.2.12 is defined as the TCLP extract. If the waste contained an initial liquid phase the filtered liquid material obtained from Sec. 7.6.2.12 and the initial liquid phase (Sec. 7.6.2.8) are collectively defined as the TCLP extract.

7.7 Documentation

7.7.1 Analysis Logbook

The analysis of samples and standards is documented within the instrument run log and supported by the instrument print-out. The runlog must be completed for each days analysis. An example of an analysis log page appears in Appendix B.

8.0 QUALITY CONTROL

NOTE: All quality control measures described in the appropriate analytical methods shall be followed.

8.1 QC Summary

8.1.1 For each batch or maximum of 20 samples extracted, an extraction blank is also extracted.

8.1.2 The blank for the non-volatile extract is two liters of the appropriate extraction fluid run through the procedure. A blank extraction fluid must be prepared for each type of fluid used per batch. If both extraction fluids are used, two blanks must be analyzed. The blank for the volatile analysis is the ZHE vessel filled with the extraction fluid and run through the procedure.

8.1.3 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. The extraction fluid is to be made up daily and the pH determined and recorded within the acceptable limits.

8.1.4 A matrix spike shall be performed for each waste type (e.g. wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.1.5 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.1.6 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.1.7 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2 Corrective Action

Since this is a preparation step, problems will not be known until the filtrates are analyzed. Corrective action for poor blank results will require all samples in the set to be re-prepared. Refer to the analytical SOPs for corrective actions.

9.0 DATA ANALYSIS AND CALCULATIONS

Since this is a preparatory procedure, refer to the analytical SOPs for matrix and method QC calculations.

9.1 Multiphasic Wastes with Non-compatible Liquid Phases

Determine the volume of the individual phases, analyze as appropriate, and combine the results mathematically by using a volume weighted average:

$$\text{Final Analyte Conc.} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = Volume in first phase (L)
V_2 = Volume in second phase (L)
C_1 = Conc. in first phase (mg/L)
C_2 = Conc. in second phase (mg/L)

10.0 WASTE MANAGEMENT AND POLLUTION CONTROL

Refer to the SOP entitled "Disposal of Laboratory Waste".

11.0 METHOD PERFORMANCE CRITERIA

Refer to section 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Figure 1. TCLP Flowchart
Table 1. TCLP Constituents and Regulatory Levels
Appendix A. TCLP Metals Spiking
Appendix B. TCLP Extraction Log

Historical File: Revision 00: 03/21/91 Revision 06: 05/05/00
Revision 01: 06/19/92 Revision 07: 05/25/01
Revision 02: 08/17/93
Revision 03: 11/03/94
Revision 04: 10/22/96
Revision 05: 03/30/99

Reasons for Change, Revision 07:
- Annual Review – No Changes.

U:\QC\SOP\SP\1311.DOC

Figure 1.

TCLP Flowchart

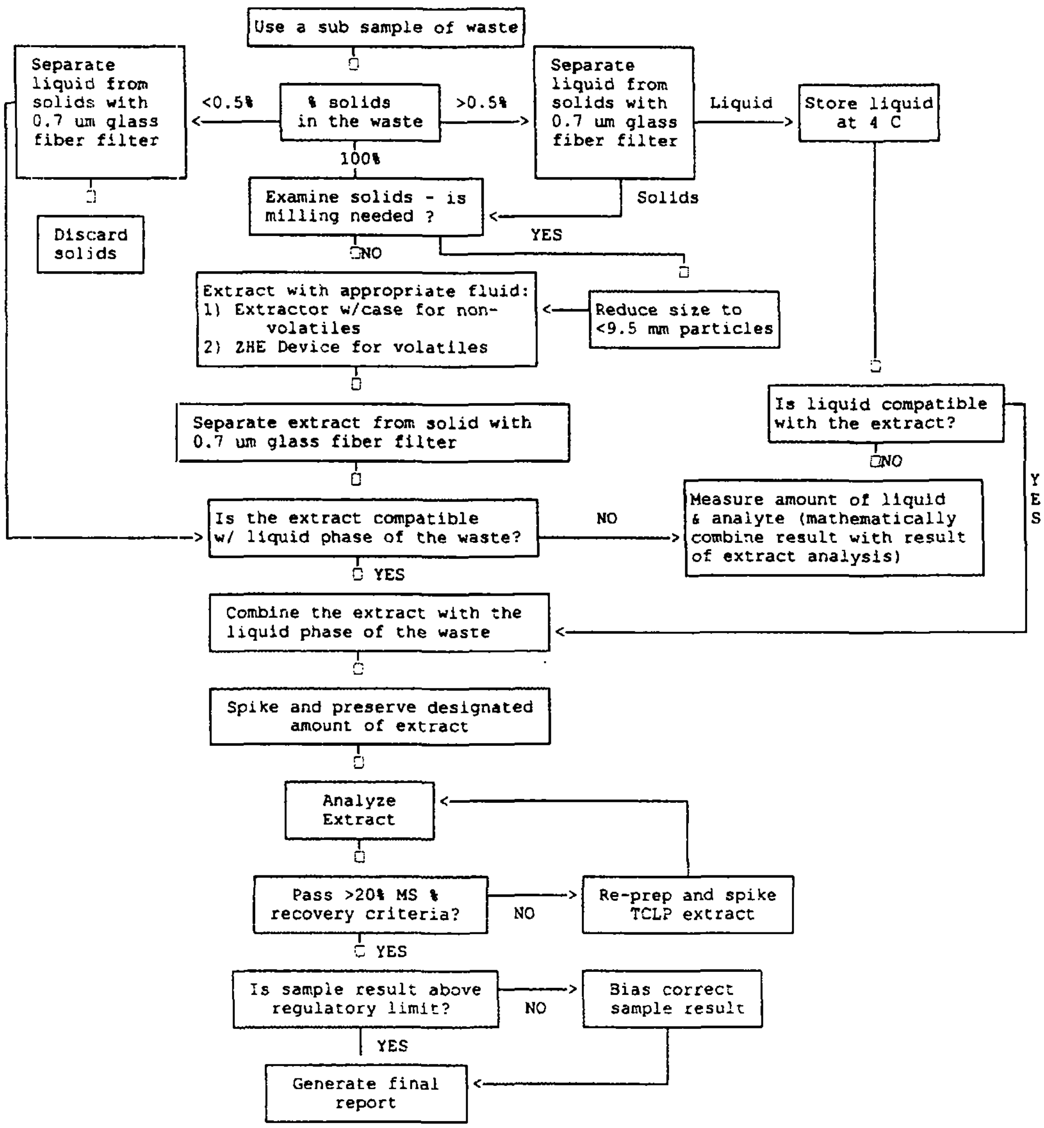

Table 1.

TCLP Constituents and Regulatory Levels

EPA HW Number	Constituent	CAS No.	Regulatory Level (ug/L)
D004	Arsenic	7740-38-2	5,000
D005	Barium	7440-39-3	100,000
D018	Benzene	71-43-2	500
D006	Cadmium	7440-43-9	1,000
D019	Carbon Tetrachloride	56-23-5	500
D020	Chlordane	57-74-9	30
D021	Chlorobenzene	108-90-7	100,000
D022	Chloroform	67-66-3	6,000
D007	Chromium	7440-47-3	5,000
D023	o-Cresol	95-48-7	*[1]200,000
D024	m-Cresol	108-39-4	*[1]200,000
D025	p-Cresol	108-44-5	*[1]200,000
D026	Cresol		*[1]200,000
D016	2,4-D	94-75-7	10,000
D027	1,4-Dichlorobenzene	106-46-7	7,500
D028	1,2-Dichloroethane	107-06-2	500
D029	1,1-Dichloroethylene	75-35-4	700
D030	2,4-Dinitrotoluene	121-14-2	130
D012	Endrin	72-20-8	20
D013	Heptachlor (& its epoxides)	76-44-8	8
D032	Hexachlorobenzene	118-74-1	130
D033	Hexachloro-1,3-butadiene	87-68-3	500
D034	Hexachloroethane	67-72-1	3,000
D008	Lead	7439-92-1	5,000
D013	Lindane	58-89-9	400
D004	Mercury	7439-97-6	200
D014	Methoxychlor	72-43-5	10,000
D035	Methyl Ethyl Ketone (2-Butanone)	78-93-3	200,000
D036	Nitrobenzene	98-95-3	2,000
D037	Pentachlorophenol	87-86-5	100,000
D038	Pyridine	110-86-1	5,000
D010	Selenium	7782-49-2	1,000
D011	Silver	7740-22-4	5,000
D039	Tetrachloroethylene	127-18-4	700
D015	Toxaphene	9001-35-2	500
D040	Trichloroethylene	79-01-6	500
D041	2,4,5-Trichlorophenol	95-95-4	400,000
D042	2,4,6-Trichlorophenol	88-06-2	2,000
D017	2,4,5-TP (Silvex)	93-72-1	1,000
D043	Vinyl Chloride	75-01-4	200

[1] If o-, m-, p-cresol concentration cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level for total cresol is 200, 000 ug/L.

Appendix A.

TCLP Metals Spiking

The purpose of the matrix spike is to monitor the performance of the analytical methods used and to determine whether matrix interferences exist.

Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to the TCLP extraction of the sample.

In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of the TCLP extract as that which was analyzed for the unspiked sample.

The following steps detail the TCLP metals spiking procedure:

- Measure out 100 mLs of TCLP extract and transfer it into a small container.
- Using an eppendorf pipet, dispense 1 mL of each standard, TCLP-1 and TCLP-2, into the TCLP extract.
- Preserve the TCLP spiked extract with 2 mLs of concentrated nitric acid.
- Store at 4 $\pm$ 2°C.

NOTE:

TCLP Stock Spike Solution Concentration:
Ba = 1000 ppm; As, Cr, Pb = 500 ppm; Cd, Se, Ag = 100 ppm.

Element Concentrations in Spiked Samples:
Ba = 100 ppm; As, Cr, Pb = 5 ppm; Cd, Se, Ag = 1 ppm

Appendix B.

Example: TCLP Extraction Logbook

STL Chicago
TCLP Extraction Logbook

Page Number:____________

Rotator Checked:
Group Number:__________________
LabNet Batch No.:________________
Sample Size Specifications:____<9.5 mm_____

Extraction Start Date / Time:_________________
Extraction Start Temperature:____________°C__
Extraction End Date / Time:_________________
Extraction End Temperature:____________°C__

Filtration Start Time:__________
Filtration End Time:__________
ZHE Initial: ___________psi
ZHE Final: ___________psi

Sample Number					
Sample Description					
Sample Weight (g)					
Liquid-Solid Separation (Yes/No)					
Volume of Mother Liquid (mLs)					
Solid Extraction Material (g)					
Extraction Fluid Selection pH of Initial Solution: If <5.0, use Extraction Fluid #1					
pH of Acid/Heat Treated Solution: If <5.0, use Extraction Fluid #1 If >5.0, use Extraction Fluid #2					
Extraction Fluid Type (1 or 2)					
Extraction Vessel Type / Pressure Check					
Extraction Fluid Volume (mLs)					
Extract Filtered (Yes or No)					
Mother Liquid Added (mLs)					
Combined Filtrate Volume (mLs)					
Final pH Reading					
Spike Solution Added (mLs)					
Spike Source ID #					
Filtrate Preserved					

Comments:____

Extraction Vessel Codes: T = Teflon; Organics/Metals | ZHE = Zero Headspace VOA's | HDPE = High Density Polyethylene Metals

Analyst: ______________________________________ Date:____________________

Reviewer:______________________________________ Date:____________________

STL CHICAGO
LABORATORY STANDARD OPERATING PROCEDURES

SOP No.	Revision No.	Date	Page
UGE-DRO	05	02/15/02	1 of 21

TITLE: Gas Chromatography: Semi-Volatiles Diesel Range Organics (DRO)

Updated by:	Signature:	Date:
Linda S. Mackley Section Manager, Organics Dept.	Linda S Mackley	2-6-02

Approved by:	Signature:	Date:
Linda S. Mackley Section Manager, Organics Dept.	Linda S Mackley	2-6-02
David L. Kaczka Env. Health & Safety Coor.	David L Kaczka	2-6-02
Terese A. Preston Quality Manager	Terese A. Preston	2-8-02

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ISSUED TO : Mark Densmore, Secor
Re: UTC Proposal

Full Signature Approvals Are Kept on File
with STL's QA Standard Practice Records

1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) provides gas chromatographic conditions for the detection of diesel range organics (DRO). Unless otherwise specified, hydrocarbons eluting from C10 (decane) to C28 (octocosane) are quantitated using diesel fuel composite standard. Narrower and wider hydrocarbon ranges may also be used. An alkane standard ranging from C8 through C36 (even only) is analyzed with each initial calibration. Quantitation using standards other than Diesel Fuel are possible, but are addressed on a case by case basis.

This SOP has been written based on SW-846 Method 8015B, all associated SW-846 methods, and the California Department of Health Services (DHS) Total Petroleum Hydrocarbon (TPH) as references.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants". MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually.

1.1.2 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. Wherever possible, reporting is limited to values approximately 2-5x the respective MDL to ensure confidence in the value reported.

Attachment 1 defines the laboratory's reporting limits and the statistically-derived control limits.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Manual (LQM, Revision 01).

1.2 Summary of Method

Prior to analysis, samples must be extracted using the appropriate techniques (refer to Section 7.3). The extracts are analyzed by direct injection into a gas chromatograph (GC). A GC utilizing a temperature program is used to separate the organic compounds and detection is achieved using a flame ionization detector (FID). This method provides the GC conditions and necessary standardization procedures for the detection of ppm levels of diesel range organics.

2.0 INTERFERENCES

- Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

3.0 SAFETY

- Employees will adhere to the practices and policies in the STL Corporate Safety Manual (CSM) and will read the MSDSs for the materials used in this method before handling or using the material.
- Interior parts of GC's can be very hot. Care should be taken if adjusting instrument.

4.0 EQUIPMENT AND SUPPLIES

4.1 Gas Chromatographs

- Hewlett-Packard 5890 Gas Chromatograph with Flame Ionization Detector (FID) and 7673 Automatic Sampler.

4.2 Columns

- Xti-5 30 M long 0.53 mm ID and 0.5-micron film thickness or equivalent is used for the analysis.

4.3 Instrument Conditions (Conditions may be altered to improve resolution.)

Carrier Gas:	UHP Helium	Detector Temp.:	300°C
Initial Temp.:	50°C	Injector Temp.:	280°C
Initial Hold:	5 minutes	Total Time:	40 minutes
Ramp Rate:	12.5°C/min	Final Hold:	15 minutes
Final Temp.:	300°C		

Each GC uses TurboChrom for data acquisition and Target for processing data.

4.4 Pipettes/Volumetric Flasks

- Micro eppendorf pipette
- Class "A" volumetric pipets and flasks of various volumes.

5.0 REAGENTS AND STANDARDS

5.1 Reagents

5.1.1 Methylene Chloride - Pesticide Grade or better
Methanol - Pesticide Grade or better
Acetone - Pesticide Grade of better

5.2 Quality Control (QC) Solutions

5.2.1 Surrogate Spike Solution

2-Fluorobiphenyl and o-Terphenyl are used as the surrogate compounds for DRO analysis. The desired final concentration of the surrogate spike is 200 ug/mL in acetone. 0.5 mL of this solution is added to all samples, spikes, QC samples and blanks prior to extraction. Surrogates are also added to calibration standards (refer to Section 5.2.3.1).

- Label Information: The label must contain the date prepared, the date of expiration, the analyst, and the standard number. All standards and spikes must be stored in Teflon-sealed screw-cap bottles with minimal headspace at 4 $\pm$ 2°C and protected from light.

5.2.2 Spike Solution

The spike solution consists of diesel fuels at 4,000 ug/mL in methanol. 0.5 mLs of spike solution is added to matrix spikes (MS) and laboratory control sample (LCS) before extraction. The stock standard solutions must be replaced after 6 months or sooner if comparison with check standards indicates a problem. However, diesel fuel reference materials may vary slightly from vendor to vendor.

- Label Information: The label must contain the date prepared, the date of expiration, the analyst, and the standard number. All standards and spikes must be stored in Teflon-sealed screw-cap bottles with minimal headspace at 4 $\pm$ 2°C and protected from light.

5.2.3 Standards

Purchased from vendors such as Restek, NSI or Absolute in solutions at concentrations ~100 times higher than the highest calibration standard.

5.2.3.1 Calibration Standards

Calibration standards at a minimum of five concentration levels for each parameter of interest are prepared through dilution of the stock standards with Methylene chloride. One of the concentration levels should be at a concentration equivalent to, or below the reporting limit. The remaining concentration levels define the working range of the GC. Calibration solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

Currently, diesel fuel standards are run at concentrations of 25, 100, 250, 500, 750 and 1000 ng/uL (Attachment 2). This is subject to change as instrument conditions change. Reporting limits are based on the lowest point of the calibration curve. Surrogate compounds are also added to the calibration standards. The levels of the surrogates in the 6 calibration standards are currently 1.0, 5.0, 10, 25, 35 and 50 ng/uL, respectively.

- Label Information: The label must contain the date prepared, the date of expiration, the analyst, and the standard number. All standards and spikes must be stored in Teflon-sealed screw-cap bottles with minimal headspace at 4 $\pm$ 2°C and protected from light.

Alkane standard – A standard containing a homologous series of n-alkanes is used for establishing retention times (C8 through C36, even only).

5.2.3.2 Second Source Verification Standard (SSV): 250 ug/mL

The SSV is a mid-level standard prepared from a second-source standard purchased from a vendor (i.e., Restek or NSI). The concentration is consistent with the cited Diesel 250 concentration listed in Attachment 2.

- Label Information: The label must contain the date prepared, the date of expiration, the analyst, and the standard number. All standards and spikes must be stored in Teflon-sealed screw-cap bottles with minimal headspace at 4 $\pm$ 2°C and protected from light. This solution is valid for 6-months.

5.2.3.3 Continuing Calibration Verification Standard (CCV): 250 & 500 ug/mL

The CCVs consisting of two concentrations are prepared independently, but from the same source, as the calibration standards (usually NSI). The concentrations are consistent with the cited Diesel 250 & 500 concentrations listed in Attachment 2. These two concentrations are alternated throughout the analytical sequence.

- Label Information: The label must contain the date prepared, the date of expiration, the analyst, and the standard number. All standards and spikes must be stored in Teflon-sealed screw-cap bottles with minimal headspace at 4 ± 2°C and protected from light. This solution is valid for 6-months.

6.0 CALIBRATION - NON-DAILY

NOTE: All standards and samples must be allowed to reach room temperature prior to analysis.

6.1 Since this method compares a diesel fuel standard to all hydrocarbons within a range, the total areas (not heights) of all peaks are used for quantitation. Each day, a standard containing the first and last hydrocarbons of the range used (i.e., C10 and C28) are run to determine the proper range. Again, some clients may request a specific range, for example C10 through C34. It is important to know this prior to running samples so that all standards and samples are quantitated the correct way. All peak areas within the specified range, with the exception of the surrogates, are added together. The total area of the diesel fuel standard is compared to the total area of the samples.

6.2 Prepare at least 5 levels of calibration standards of Diesel Fuel and the surrogates from the concentrated stock (Section 5.2.3). The calibration standards should range from the lowest, being at or below the reporting limit, through to the highest which should define the working range of the GC.

6.3 Inject each calibration standard using the same sample introduction technique that will be used to introduce the actual samples. The ratio of response to the amount injected, defined as the calibration factor (CF), can be calculated for each analyte at each standard concentration. If the percent relative standard deviation (%RSD) of the calibration factors is less than or equal to 20% over the working range, linearity through the origin can be assumed and the average calibration factor can be used for calculations.

$$\text{Calibration Factor} = \frac{\text{Total Area of Peaks}}{\text{Mass Injected (in nanograms)}}$$

$$\%RSD = \frac{\text{Standard Deviation of CFs}}{\text{Average CF}} \times 100$$

6.4 The calibration curve must be verified on each working day, and after every 10 samples, by injecting a mid-level calibration standard as continuing calibration verification (CCV). If the response for the CCV varies from the predicted response by more than $\pm$15%, a new calibration sequence must be analyzed.

$$\% \text{ Difference} = \frac{R_2 - R_1}{R_1} \times 100$$

Where:
R_1 = Average CF from linearity
R_2 = CF from succeeding analyses

6.5 Hydrocarbon Range

6.5.1 DROs are quantitated using hydrocarbon peaks (excluding surrogates) eluting between an initial and final peak. Typically, the C10 (decane) through C28 (octocosane) peaks are used. Narrower or wider ranges may also be used.

6.5.2 The retention times of the initial and final hydrocarbon of the range are determined by running a component standard containing both hydrocarbons prior to all samples. The range should include these hydrocarbons.

6.5.3 The retention times of the surrogate compounds should be monitored throughout the analysis sequence to detect shifts. If a shift occurs, the component standard should be rerun and the hydrocarbon range adjusted.

7.0 PROCEDURE

7.1 Quality Control Checks

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 samples	< Rpt. Limit
Lab Control Sample (LCS) [1]	1 in 20 samples	Attachment 1 [3]
Matrix Spike (MS) [2]	1 in 20 samples	Attachment 1 [3]
MS Duplicate (MSD) [2]	1 in 20 samples	Attachment 1 [3]
Surrogate	every sample/MB/LCS/MS/MSD	Attachment 1 [3]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is random, unless specifically requested by a client.
[3] Statistical control limits are updated annually.

7.2 Sample Preservation and Storage

Water and soil samples must be collected in glass containers with Teflon-lined lids. If Teflon-lined lids are not available, aluminum foil should be placed between the sample and the lid with the dull side of the foil towards the sample. Samples and extracts are stored at 4 $\pm$ 2°C.

Matrix	Holding Time (VTS)[1]: to Extract	Holding Time: To Analyze after Extraction	Preservative[2]
Soil/Sediment	14 days	\	Cool 4 $\pm$ 2°C
Water	7 days	40 days	Cool 4 $\pm$ 2°C
Waste/Oil	14 days	/	Cool 4 $\pm$ 2°C

[1] VTS = Verified Time of Sampling
[2] Prior to extraction; after extraction (prior to analysis).

7.3 Sample Preparation

The sample matrix determines which extraction procedure to follow. Waters are extracted following the separatory funnel (SOP No. USP-3510) method; soils are by automated soxhlet extractor (USP-3541) or sonication (USP-3550); and wastes and oils are by dilution (USP-3580). Refer to the specific SOPs for the extraction procedures.

7.4 Calibration / Standardization - Daily

NOTE: All standards and samples must be allowed to reach room temperature prior to analysis.

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

Calibration Controls	Sequence	Control Limit
Alkane standard	Prior to initial calibration	Establish retention time range.
Initial Calibration	5-pt. (min) linearity	< 20% RSD
Second Source Verif (SSV)	Following ICAL	$\pm$ 15% pred. rsp.
Cont. Cal. Verif. (CCV)	prior to and after every 10 injections	$\pm$ 15% pred. rsp.

7.5 Preventive Maintenance

- The septum should be changed between each analysis sequence. No more than 100 injections should be made without changing the septum.
- Disposable glass injector insert should be changed when it becomes discolored.

- Periodically the entire system should be checked for leaks and frayed wires.
- See the instrument manuals if problems are encountered which cannot be resolved by routine maintenance.

7.5.1 Suggested Maintenance

When any of the criteria described in Sections 7.1, 7.4, and 8.1 is out of control, one or more of the following actions may be necessary:

- Change the septum.
- Change disposable glass injector insert if discolored.
- Remove approximately 12 inches from the front of the column.
- Check and adjust all flows.
- Bake the injector, oven or detector at approximately 20°C above normal.

7.6 Gas Chromatographic Analysis

7.6.1 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins the initial calibration using at least 5 levels of standards. The sequence continues with the analysis of a SSV and CCV. If comparison of the CF from the SSV and CCV is within ±15% of the calibration standards average CF then the analysis sequence may proceed with the MB, followed by samples (if the blank is good). If the CF of any CCV is greater than ±15% difference, then the standard is re-analyzed. If the CF is still greater than ±15% difference, a new calibration sequence must be analyzed.

7.6.2 The CCV must be injected after every 10 injections. The calibration factor for each CCV must not exceed a 15% difference when compared to the average CF from the initial calibration sequence. When this criterion is exceeded, inspect the GC system to determine the cause and perform whatever maintenance necessary before re-analyzing the standard. If the CF's still exceed the 15% difference, a new calibration sequence is required and all samples that were injected after the last good CCV must be re-injected.

7.6.3 A MS/MSD is performed every 20 samples on a randomly chosen sample. After that sample is analyzed, the MS and MSD are analyzed. Also, the LCSs are analyzed following the analysis of the MB. The sequence ends when all samples are analyzed or when qualitative and/or quantitative QC criteria are exceeded.

7.6.4 If the responses exceed the linear range of the system, dilute the sample and re-analyze. It is recommended that the samples be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, is acceptable if linearity is demonstrated.

7.6.5 Identification occurs whenever any peaks elute within the specified range. All peaks, regardless if they match the standard pattern or not, are used for quantitation. All hydrocarbons will be quantitated against the diesel fuel standards and reported as "diesel range organics".

7.6.6 Validate the qualitative performance of the GC system by running the CCVs throughout the analysis sequence to evaluate this criterion. If either of the surrogates in a standard fall outside their daily retention time window, the system is out of control. Determine the cause of the problem and correct it.

7.6.7 Retention Time Windows – established for surrogates.

7.6.7.1 The retention time ***range*** for DROs is defined during initial calibration. The range is established from the retention times of the C10 and C28 alkanes (if a narrower or wider range is requested the appropriate alkanes would be used). DRO is distinguished on the basis of the ranges of retention times for characteristic components of the fuel.

7.6.7.2 Before establishing windows for the surrogate compounds, make sure the GC system is within optimum operating conditions. Make three (3) injections of the standard mixture throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

7.6.7.3 Record the retention time for the surrogates to three decimal places. Calculate the mean and the standard deviation of the three absolute retention times.

7.6.7.4 If the standard deviation of the retention times is 0.000 (no difference between the three retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

7.6.7.5 The width of the retention time window is defined as plus or minus three times the standard deviation of the absolute retention times. If the default standard deviation is employed, the width of the window will be 0.03 minutes.

7.6.7.6 Establish the center of the retention time windows from the calibration verification at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. Retention time windows can be updated every 12 hours. However, they are usually only updated at the onset of a continuing calibration sequence or after maintenance has been performed.

7.6.7.7 The laboratory must calculate retention time windows for each surrogate on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory and available for review.

7.6.8 Manual Integration Policy

In each case where manual integrations have taken place, the operator must identify, initial and date the changes on the hardcopy. The following guidelines apply with complete details in the Manual Integration SOP (UQA-037):

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.

Manual integrations are most often performed for the following reasons. If a manual integration is performed for a reason other than listed, the reason will be documented and approved by the section manager.

- Assignment of correct peak that was mis-identified by the system.
- Incomplete auto-integration due to high level of target detected.
- Incomplete auto-integration due to background interference.
- Incorrect auto-integration due to co-elution or near co-elution of compounds.
- Missed peaks.

All integrations are reviewed by the analyst. All chromatograms and reports are printed after any integrations take place and are routinely included in the data packages. Manual integrations may be documented in the narrative if so required, however, references to this SOP will be used for explanations, and any further documentation beyond initials and dates will not be done.

7.7 Documentation

7.7.1 Instrument Run-Logs

The analysis of samples and standards is documented within each instrument-specific run log (Attachment 3) and must be completed for each days analysis.

7.7.2 Traceability of Standards

When a run log is set up for each instrument, all initial standards are noted in the logbook with the standard #'s. This allows for traceability of the original standard. It is assumed that if no further notations are made in the runlog concerning the standard identification, book #'s of the initial standards used will be the same standard throughout the analytical sequence.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. The analyst and a trained data reviewer perform the reviews according to the criteria established on the data review form (Attachment 4). Upon the first 100% review, the review form is initialed and dated as reviewed. The package, with its review sheet, comments and any corrective action reports is submitted to the unit leader, section manager, or peer reviewer for a second review. Once again, the review form is initialed and dated by the second reviewer. The completed data review form remains on file with the original data.

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 At least one MB and one LCS will be included in each laboratory batch of 20 samples. The MBs will be examined to determine if contamination is being introduced in the laboratory. The LCS will be examined to determine both precision and accuracy.

8.1.2 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

8.1.3 Precision will be measured by the reproducibility of the MSs and will be calculated as Relative Percent Difference (RPD). If MSs were not analyzed, reproducibility will be measured using the LCS/LCD. Results must agree within statistical control limits in order to be considered acceptable.

8.1.4 Surrogate compounds will be added to every sample to measure performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable.

8.1.5 For each analytical batch (20 samples), a MB, LCS and MS/MSD must be analyzed. The blank and spike samples must be carried through all stages of the sample preparation and measurement steps.

8.1.6 Each time an analytical sequence is started the standard solution must be evaluated to determine if the chromatographic system is operating properly. The analyst should consider--Do the peaks look normal?, Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc.

8.1.7 The laboratory must maintain records to document the quality of the data generated. When results of sample spikes indicate irregular method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.8 Before analysis of any samples, the analyst should demonstrate, through the analysis of a MB, that interferences from the analytical system, glassware and reagents are under control.

8.1.9 Each day that analysis is performed, the daily calibration sample should be measured to determine if the chromatographic system is operating properly. If any changes are made to the chromatographic system, recalibration of the system must take place.

8.1.10 Required Instrument QC

8.1.10.1 The method requires that the %RSD vary by <20% when comparing calibration factors to determine if the initial calibration standards are linear through the origin.

8.1.10.2 The method sets a limit of $\pm$15% difference when comparing the continuing response of a given analyte versus the initial response. If the limit is exceeded corrective action must be taken to correct the problem, or the sequence must be started over. All samples following the last standard that was in-control must be reanalyzed.

8.1.10.3 For every batch of samples (20 samples = a batch) the analyst must perform a MB, LCS, MS/MSD. Also, every sample, spike and blank must be spiked with the surrogate solution. Limits used for spike recoveries are in-house generated control limits, or limits which have been specifically assigned by the client. See your backlog for QC type requested.

8.2 Corrective Actions

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources when determining the action to be taken. The out of control situation may or may not be caused by more than one problem. The analyst should seek the help of his/her immediate supervisor, QA personnel, or other experienced staff if they are uncertain of the cause of the out of control situation and the corrective action. The analysis must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the supervisor, QA personnel or the laboratory manager.

Listed below are steps that must be taken when an out of control situation occurs:

- Demonstrate that all the problems creating the out of control situation were addressed;
- Document the problem and the action that was taken to correct the problem on a corrective action report form;
- Document on the corrective action report that an in control situation has been achieved; and
- Receive approval (signature) of the Section Manager, QA personnel, or the laboratory manager prior to the release of any analytical data associated with the problem.

Whenever a problem exists, such as insufficient sample to run a MS/MSD, a Sample Discrepancy Report (SDR) is written. It is filed with the report discussing the actions taken to correct and document the problem. The analyst and their Section Manager decide what to do with this problem, whether it is analytical, sampling, or matrix interference.

Through out Sections 7 and 8, numerous criteria are described which must be met to meet analytical requirements. Listed below are some suggested courses of action that may be taken to correct out of control situations that may occur with the procedure.

8.2.1 Calibration Curve

- Reanalyze the standard curve.
- Prepare new stock and/or working standards.

8.2.2 Continuing Calibration Verification (CCV)

- Repeat the CCV to verify proper preparation.
- Prepare a new CCV from original stock.
- Check for instrument drift.
- Recalibrate with new standard curve and repeat all samples since the previous in control CCV.
- Prepare new stock and/or working standards.

8.2.3 Laboratory Control Sample (LCS)

If LCS is low -

- Determine the source of the error within the sample preparation and repeat the set, **WRITE A CAR**.

write a car

If LCS is high -

- Check for source of contamination.
- Correct for contamination and repeat set, **WRITE A CAR**.

8.2.4 Laboratory Control Sample Duplicate (LCD)

- The LCD must meet all control limits as LCS in addition to limits set for precision (same corrective action as LCS).

8.2.5 Method Blank (MB)

- Reanalyze the MB to verify that it is beyond detection limit.
- Check and correct for any source of contamination and repeat sample set, **WRITE A CAR**.
- In the extreme case where all samples in the set are at least 10 times greater than the MB, reanalyses may not be required, **WRITE A CAR AND GET SUPERVISORS APPROVAL.**

8.2.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

- If both the MS and MSD recoveries are low or high (RPD within control), sample matrix may be explained for the low/high recoveries; re-analysis is recommended.
- Regardless of the out come of the reanalysis, a **CAR** will be written and approved.

8.2.7 Surrogate Recovery

- If surrogate recoveries are biased high, evaluate the chromatogram and determine if it is due to the level of DRO or interferences present in the sample. Write a CAR and address the situation in the case narrative.
- Check to be sure that there are no errors in the calculations or surrogate solutions.
- Check the instruments performance. If a problem is identified with the instrument, correct the problem and re-analyze the extracts.
- If no instrument problem is found, the sample should be re-extracted and re-analyzed. An SDR must be initiated so that the PM, Section Manager and client are notified of the situation. If the holding time for the extraction has expired, report both sets of data. Note in the narrative if the holding times were expired, if surrogate recoveries were still outside of control, or if the re-extract provided acceptable recoveries.
- If surrogate recoveries are high and the sample is non-detect for DRO, write a CAR. Re-extraction may be necessary if required by client. In some instances when the surrogates are high and the sample is non-detect, no further action will be required. Consult with the Section Manager or Project Manager to determine action required.
- If surrogate recoveries are low and an MS/MSD were performed on the sample with low recovery, and both the MS and MSD also have low surrogate recoveries, matrix may be the cause of the low recoveries. Document in a CAR for inclusion in the case narrative.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Sample Concentration

The concentration of each analyte in the sample may be determined using the following calculations:

$$\frac{(As)(Vt)(D)}{(CF_{avg})(Vs)} = \text{concentration of DRO in sample (mg/L or mg/Kg)}$$

Where:
As = Area of peaks in sample
Vt = Total volume of the concentrated extract (uL)
D = Dilution factor (D=1 if no dilution is made)
CF_{avg} = Mean calibration factor from the initial calibration (area per ng.)
Vs = Volume of sample or weight of sample extracted

9.2 Dry Weight

All soil samples must be reported on a "dry weight" basis:

$$\frac{\text{"As is" Conc. in sample}}{\text{\% Total Solids (in decimal form)}} \times 100 = \text{dry weight conc. in sample}$$

9.3 % Recovery (surrogate) = $\frac{SR}{SA} \times 100$

Where:
SR = Surrogate result determined from the analysis.
SA = Surrogate added.

9.4 % Recovery (spikes) = $\frac{Cs - Cu}{Cn} \times 100$

Where:
Cs = Measured conc. of spiked sample aliquot.
Cu = Measured conc. of unspiked sample aliquot (0 for LCS).
Cn = Nominal conc. increase that results from spiking the sample, or the nominal concentration of the spiked aliquot (for LCS).

9.5 Precision = $\frac{|C1 - C2|}{\frac{C1 + C2}{2}} \times 100$

Where:
C1 = Measured concentration of the first sample aliquot.
C2 = Measured concentration of the second sample aliquot.

9.6 **% Moisture** = 100 - % Total Solids*

* % Total Solids are performed by the Metals Department (USP-2540G).

10.0 WASTE MANAGEMENT AND POLLUTION CONTROL

Waste from this procedure will enter the "Flammable liquids in vials" wastestream. Single component standards will be turned over to the EHSC or Waste Technician.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Attachment 1. Example: Laboratory Reporting and Control Limits
Attachment 2. Example: DRO Standard Concentrations
Attachment 3. Example: Analysis Run Log
Attachment 4. Example: Data Review Form

Historical File: Revision 00: 02/16/95
Revision 01: 12/07/95
Revision 02: 02/16/98
Revision 03: 03/23/99
Revision 04: 09/28/00
Revision 05: 02/06/02

Reasons for Change, Revision 05:

- Clarification of retention time windows for surrogates and retention time range for DRO (Sec. 7.6.6 and 7.6.7)
- Addition of SSV and CCV sections for clarification – alternating concentrations of CCVs (Sections 5.2.3.2 & 5.2.3.3).

U:\QC\SOP\GE\DRO.DOC

Attachment 1.

Example: Laboratory Reporting and Control Limits

Note:
Reporting limits will vary depending on sample/limited sample size/volume, dilution factors, dry weight adjustment for total solids.

STL Chicago
Method Limit Report for SW-846 Method 8015B (Diesel Fuel) Date: 1/28/02

Description	Matrix	Units	MDL[1]	Reporting Limit[2]	Lab Control Standard[3]			Surrogates[3]	
					Lower Limit	Upper Limit	RPD	Lower Limit	Upper Limit
TPH - Jet Fuel (JP4)	Water	mg/L	0.125	0.125	63	107	20		
TPH - Jet Fuel (JP5)	Water	mg/L	0.125	0.125					
TPH - Jet Fuel (JP8)	Water	mg/L	0.125	0.125					
Stoddard Solvents	Water	mg/L	0.125	0.125					
Diesel Range Organics (DRO)	Water	mg/L	0.086	0.125	63	107	20		
Motor Oil (MRO)	Water	mg/L	0.124	0.25					
Total TPH	Water	mg/L	0.125	0.125					
TPH - Jet Fuel (JP4)	Oil	mg/Kg			72	120	20		
TPH - Jet Fuel (JP5)	Oil	mg/Kg							
Stoddard Solvents	Oil	mg/Kg							
Diesel Range Organics (DRO)	Oil	mg/Kg	250	250	72	120	20		
Motor Oil (MRO)	Oil	mg/Kg	500	500					
TPH - Jet Fuel (JP4)	Solid	mg/Kg	4.2	4.2	72	120	20		
TPH - Jet Fuel (JP5)	Solid	mg/Kg	4.2	4.2					
TPH - Jet Fuel (JP8)	Solid	mg/Kg	4.2	4.2					
Stoddard Solvents	Solid	mg/Kg	4.2	4.2					
Diesel Range Organics (DRO)	Solid	mg/Kg	3.2	4.2	72	120	20		
Motor Oil (MRO)	Solid	mg/Kg	8.3	8.3					
Total TPH	Solid	mg/Kg	4.2	4.2					
TPH - Jet Fuel (JP4)	3541	mg/Kg	4.2	4.2	72	120	20		
TPH - Jet Fuel (JP5)	3541	mg/Kg	4.2	4.2					
TPH - Jet Fuel (JP8)	3541	mg/Kg	4.2	4.2					
Stoddard Solvents	3541	mg/Kg	4.2	4.2					
Diesel Range Organics (DRO)	3541	mg/Kg	2.6	4.2	72	120	20		
Motor Oil (MRO)	3541	mg/Kg	8.3	8.3					
Total TPH	3541	mg/Kg	4.2	4.2					
Surrogates									
2-Fluorobiphenyl (surr)	Water	mg/L						25	129
o-Terphenyl (surr)	Water	mg/L						37	159
2-Fluorobiphenyl (surr)	Oil	mg/Kg						33	115
o-Terphenyl (surr)	Oil	mg/Kg						34	168
2-Fluorobiphenyl (surr)	Solid	mg/Kg						33	115
o-Terphenyl (surr)	Solid	mg/Kg						34	168
2-Fluorobiphenyl (surr)	3541	mg/Kg						33	115
o-Terphenyl (surr)	3541	mg/Kg						34	168

Notes:

1 MDLs: Are determined on an annual basis and are subject to change. Contact the laboratory for the most current values.

2 RLs: Will vary depending on MDLs; sample volume/size; dilution factors; and dry weight reporting (solids).

3 LCS & Surrogate Control Limits: Are tabulated annually. Contact the laboratory for the most current limits.
LCS limits are also applicable to matrix QC (i.e., MS/MSDs)

Attachment 2.

Example: DRO Standard Concentrations

DRO/Diesel Fuel Standard Concentrations

Calibration Standards	Diesel 25	Diesel 100	Diesel 250	Diesel 500	Diesel 750	Diesel 1000	
Diesel Fuel	25	100	250	500	750	1000	µg/mL
2-Fluorobiphenyl	1.0	5.0	10	25	35	50	
o-Terphenyl	1.0	5.0	10	25	35	50	

Surrogate Concentrations	
2-Fluorobiphenyl	200 µg/mL
o-Terphenyl	200

Spike Concentration	
Diesel Fuel	4000 µg/mL

C8-C40 RT Standard	
C8, C10, C12, C14, C16, C18, C20, C22, C24, C26, C28, C30, C32, C34, C36, C38, C40	10 µg/mL each
2-Fluorobiphenyl	10 µg/mL
o-Terphenyl	10 µg/mL

Attachment 3.

Example: Analysis Run Log

STL Chicago
GC/FID Analysis Log
Instrument 10 – HP 5890

Page #:____________

Analyst ____________________ Method:____________ Column:____________________

Queue:________________ Inj. Vol.:__________ Inj. Temp:__________ Det. Temp:__________

Temp. Program:____

Rep. #	Sample Description	Dil. Factor	Injection Date/Time	Comments

Reviewer:________________________________ Date:________________

Attachment 4.

Example: Data Review Form

STL Chicago
GC/HPLC Data Review Checklist

Project: ____________________ Job #:__________ Method:________

Reviewer (1):______________________________ Date:________

Reviewer (2):______________________________ Date:________

Sublist:__________
Instruments (Primary/Confirmation):__________
Cleanups: ______________________________
CAR (Y/N):_____

Target Review

Reviewer 1	Reviewer 2	
____	____	Chromatography is acceptable.
____	____	Chromatograms are scaled properly.
____	____	All peaks are labeled properly.
____	____	All initial calibrations are within control limits ($\leq$ 20% RSD; Correlation Coefficient $\leq$ 0.995).
____	____	Second Source Verification is in control (85% - 115%).
____	____	All continuing calibrations are within control limits ($\pm$ 15% difference).
____	____	All retention times are within their windows.
____	____	All method blanks are clean.
____	____	Calculations verified.
____	____	Verify samples are quantified using the proper ICAL.
____	____	Before and after chromatograms produced for all manual integrations.

Comments:______________________________

Updating Results in LabNet

____	____	Reagent codes are correct.
____	____	Batch test results match quant reports.
____	____	Batch cloned for project limits.
____	____	Proper prep links were created (including TCLP link).
____	____	Each required target compound diplays "0" when data is reported.
____	____	Client information has been checked for correct list, reporting limits, special requirements.
____	____	Job notes (CTRL F12) for the job have been reviewed.

Comments:______________________________

QC Data

____ ____ All LCS/LCD recoveries (and RPDs) are within the required control limits (verify calc.).
____ ____ All surrogate recoveries are within the required control limits (verify calc).
____ ____ All MS/MSD recoveries and RPDs are within the required control limits (verify calc.).

Comments:____

Note: Anything out of the ordinary must be commented on and be approved by the Unit Leader/Section Manager for inclusion in the Case Narrative.

____ RG LabChron/Report Review Initial/Date ________________

Comments:____

ATTACHMENT C TO QAPP
SAMPLE LABELS AND CHAIN OF CUSTODY FORMS

SECOR Project NO.: 13UN.02072.00.0001
March 31, 2003

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

Quality Environmental Containers

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	___ GRAB ___ COMPOSITE

ANALYSIS REQUESTED

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

Quality Environmental Containers

PROJECT NAME		TARE WT
SAMPLE ID	SAMPLE DATE	SAMPLE TIME
SAMPLED BY	PRESERVATIVE	
ANALYSIS REQUESTED	___ GRAB ___ COMPOSITE	

CUSTODY SEAL

DATE ____________________

SIGNATURE ____________________

QEC
Quality Environmental Containers
800-255-3950 • 304-255-3900

SEVERN TRENT SERVICES

STL Chicago
2417 Bond Street
University Park, IL 60466
Phone: 708-534-5200
Fax: 708-534-5211

Report To:
Contact:
Company:
Address:
Phone:
Fax:
E-Mail:

To:
Contact:
Company
Address:
Phone:
Fax:
PO#:
Quote:

Shaded Areas For Internal Use ____ of ____

Lab Lot#

Package Sealed Yes No	Samples Sealed Yes No
Received on Ice Yes No	Samples Intact Yes No
Temperature °C of Cooler	
Within Hold Time Yes No	Preserv. Indicated Yes No NA
pH Check OK Yes No NA	Res Cl_2 Check OK Yes No NA
Sample Labels and COC Agree Yes No COC not present	

Sampler Name:	**Signature:**
Project Name:	**Project Number:**
Project Location:	**Date Required** Hard Copy: ___/___/___
Lab PM:	Fax: ___/___/___

Laboratory ID	MS-MSD	Client Sample ID	Sampling Date	Sampling Time	Matrix	Comp/Grab	Refrg # / # / Cont. / Volume / Preserv										Additional Analyses / Remarks

RELINQUISHED BY	COMPANY	DATE	TIME	RECEIVED BY	COMPANY	DATE	TIME
RELINQUISHED BY	COMPANY	DATE	TIME	RECEIVED BY	COMPANY	DATE	TIME

Matrix Key
WW = Wastewater
W = Water
S = Soil
SL = Sludge
MS = Miscellaneous
OL = Oil
A = Air
SE = Sediment
SO = Solid
DS = Drum Solid
DL = Drum Liquid
L = Leachate
WI = Wipe
O =

Container Key
1. Plastic
2. VOA Vial
3. Sterile Plastic
4. Amber Glass
5. Widemouth Glass
6. Other

Preservative Key
1. HCl, Cool to 4°
2. H2SO4, Cool to 4°
3. HNO3, Cool to 4°
4. NaOH, Cool to 4°
5. NaOH/Zn, Cool to 4°
6. Cool to 4°
7. None

COMMENTS

Date Received / /
Courier: **Hand Delivered** ☐
Bill of Lading

196

200

FedEx Express *USA Airbill* FedEx Tracking Number 8390 7979 4708

Form I.D. No. 0215

MUR21

1 From *Please print and press hard.*

Date ______ Sender's FedEx Account Number 2285-4050-1

Sender's Name ______ Phone (217) 698-7247

Company SECOR INTERNATIONAL INC.

Address 400 N BRUNS LN Dept/Floor/Suite/Room

City SPRINGFIELD State IL ZIP 62702-4617

2 Your Internal Billing Reference
First 24 characters will appear on invoice

3 To

Recipient's Name ______ Phone ( )

Company ______

Address ______
To "HOLD" at FedEx location, print FedEx address. We cannot deliver to P.O. boxes or P.O. ZIP codes

Address ______ Dept/Floor/Suite/Room

City ______ State ______ ZIP ______

By using this Airbill you agree to the service conditions on the back of this Airbill and in our current Service Guide, including terms that limit our liability.

Questions? Visit our Web site at fedex.com
or call 1.800.Go.FedEx® 800.463.3339.

4a Express Package Service *Packages up to 150 lbs.*
Delivery commitment may be later in some areas.

- [ ] FedEx Priority Overnight — Next business morning
- [ ] FedEx Standard Overnight — Next business afternoon
- [ ] FedEx First Overnight — Earliest next business morning delivery to select locations
- [ ] FedEx 2Day — Second business day
- [ ] FedEx Express Saver — Third business day

FedEx Envelope rate not available. Minimum charge: One-pound rate

4b Express Freight Service *Packages over 150 lbs.*
Delivery commitment may be later in some areas.

- [ ] FedEx 1Day Freight* — Next business day
- [ ] FedEx 2Day Freight — Second business day
- [ ] FedEx 3Day Freight — Third business day

* Call for Confirmation ______

5 Packaging * Declared value limit $500

- [ ] FedEx Envelope*
- [ ] FedEx Pak* — Includes FedEx Small Pak, FedEx Large Pak, and FedEx Sturdy Pak
- [ ] Other

6 Special Handling Include FedEx address in Section 3

- [ ] SATURDAY Delivery — Available ONLY for FedEx Priority Overnight and FedEx 2Day to select ZIP codes
- [ ] HOLD Weekday at FedEx Location — NOT Available for FedEx First Overnight
- [ ] HOLD Saturday at FedEx Location — Available ONLY for FedEx Priority Overnight and FedEx 2Day to select locations

Does this shipment contain dangerous goods?
One box must be checked.

- [ ] No
- [ ] Yes — As per attached Shipper's Declaration
- [ ] Yes — Shipper's Declaration not required
- [ ] Dry Ice — Dry Ice, 9, UN 1845 ______ x ______ kg

Dangerous Goods (including Dry Ice) cannot be shipped in FedEx packaging.
- [ ] Cargo Aircraft Only

7 Payment *Bill to:* Enter FedEx Acct. No. or Credit Card No. below.

- [ ] Sender — Acct. No. in Section 1 will be billed
- [ ] Recipient
- [ ] Third Party
- [ ] Credit Card
- [ ] Cash/Check

FedEx Acct. No. Credit Card No. ______ Exp. Date ______

Total Packages	Total Weight	Total Declared Value†
		$ .00

†Our liability is limited to $100 unless you declare a higher value. See back for details.

FedEx Use Only

8 Release Signature *Sign to authorize delivery without obtaining signature.*

By signing you authorize us to deliver this shipment without obtaining a signature and agree to indemnify and hold us harmless from any resulting claims.

447

0237686880

PULL AND RETAIN THIS COPY BEFORE AFFIXING TO THE PACKAGE

NO POUCH NEEDED.
See back for peel and stick application instructions.

ATTACHMENT D TO QAPP
LIST OF ACRONYMS
SECOR Project NO.: 13UN.02072.00.0001
March 31, 2003

LIST OF ACRONYMS/ABBREVIATIONS

AOC	Administrative Order on Consent
ARARs	Applicable or Relevant and Appropriate Requirements
ASTM	American Standards for Testing Materials
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act (Superfund)
COC	Chain of Custody
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limits
CRQL	Contract Required Quanitation Limits
CRL	Central Regional Laboratory
DCF	Document Control Format
DRO	Diesel Range Organics
DQO	Data Quality Objective
EAPM	Early Action Project Manager
EMSL	Environmental Monitoring and Support Laboratory
FSP	Field Sampling Plan
FSS	Field Services Section
IEPA	Illinois Environmental Protection Agency
MDLs	Method Detection Limits
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NIST	National Institute of Standard Technology
NPL	National Priorities List
QA/QC	Quality Assurance/Quality Control
QAMP	Quality Assurance Management Plan
QAPP	Quality Assurance Project Plan
QLs	Quantitation Limits
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PE	Performance Evaluation Sample
RAS	Routine Analytical Services
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation/Feasibility Study
RD/RA	Remedial Design/Remedial Action
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SAS	Special Analytical Services
SER	Southeast Rockford
SF	Superfund
SMC	Sample Management Coordinator
SOP	Standard Operating Procedure
SOW	Statement of Work
SW-846	Test Methods for Evaluating Solid Waste
TAL	Target Analytes List
TCL	Target Compound List
TSA	Technical System Audit
USEPA	United States Environmental Protection Agency

ATTACHMENT E TO QAPP
STANDARD OPERATING PROCEDURES
SECOR Project NO.: 13UN.02072.00.0001
March 31, 2003

ATTACHMENT E

TABLE OF CONTENTS

A-1

SOP FOR PRE-FIELD DRILLING ACTIVITIES

The following procedures are to be followed prior to the commencement of drilling activities at the Site:

1. Notify the appropriate state agency of intention to drill/install wells. This will often be the State Engineer's Office, but may vary by state.

   1.1 Include the following information in the Notice of Intent:
   1. Site Address/Location
   2. Site Cadastral Coordinates - Township, Range, Section
   3. Number and Type of Wells to be Drilled (i.e. 2" Groundwater Monitoring Well)
   4. Proposed Depth
   5. Land Owner
   6. Party Responsible for Wells (usually the client)
   7. Reason for Installing the Wells
   8. Proposed Drilling Date

2. Obtain local agency drilling permit(s), as necessary.

3. Obtain State Department of Transportation (or Municipality) Permits if well will be in a public right-of-way or private property access agreements if well will be on adjacent property.

4. In support of the underground utility/structure identification activities, an electromagnetic (EM) survey and/or ground penetrating radar (GPR) survey may be performed in select areas as outlined in Section 2.0 of the FSP.

5. Establish a mobile office space, sanitary facilities and communication system at the site for SECOR and USEPA.

6. The drilling contractor will arrange for utility locates. Appropriate employees from the drilling contractor and SECOR will attend locates personally. Also present at the utility locates on facility property will be a facility representative. Note overhead utilities. **Complete a signed Utilities and Structures Checklist (Attached Form) for each hole prior to drilling.**

7. Call and schedule drilling company.

   7.1 Specify well type, construction, depths, and completion details.

   7.2 Specify soil/rock sampling requirements.

8. Submit laboratory work/bottle order.

9. Review the current Health and Safety Plan (HASP) for the Site.

   9.1 Provide copy of the HASP to the drilling company in advance.
   9.2 Request proof of drilling company's OSHA safety training and medical surveillance in advance

10. Schedule required equipment and obtain needed supplies. A typical list includes:
    - Hand Auger and shovel
    - Ziplock® or equivalent sealable plastic bags
    - Paper Towels
    - Field Notebook
    - Permanent Markers/Pens
    - Water Level Indicator or Interface Probe
    - Tile probe
    - Photo-ionization Detector (PID) & Calibration Gas
    - Alconox® or similar low-phosphate cleaning agent
    - De-ionized Water
    - Nitrile Gloves
    - Disposable Bailers
    - Nylon Rope or Twine for Bailers
    - Sample containers and cooler
    - Level D Safety Equipment (Hard Hat, Boots & Safety Glasses)

**** If possible, borehole locations will be hand augured or tile probed to a depth of five feet before drilling.** (Except in areas where underground structure removal has occurred.)**

Note: In cases where hand auguring is necessary, continuous split spoon samples will also be collected to a depth of five feet within approximately 1 foot of the hand-augured hole for the purposes of sample collection. In select locations, vacuum excavation might be used.

A-2

SOP FOR SOIL BORING COMPLETION

Prior to the drilling of soil borings, scheduled drilling sites will be cleared for utilities and structures by the environmental contractor or their subcontractors. A Utilities and Structures Checklist will also be filled out and signed prior to drilling and excavation activities. A copy of this form is included in Attachment F.

Drilling will be accomplished using either a direct-push rig (Geo-Probe® type), or hollow-stem auger or air rotary or percussion drilling equipment or other applicable drilling rig capable of collecting continuous samples (split-spoon and/or dry core barrel) from the surface to the base of each hole. During drilling of soil borings, a continuous, descriptive, lithologic log will be prepared by a qualified geologist or geotechnical individual based on an examination of the split-spoon samples and soil cuttings. A copy of a blank boring log form is included in Attachment F. In the event that the soil boring can not be completed to a satisfactory depth, an alternative site may be chosen.

Continuous split-spoon samples will be obtained at each soil boring. The soil cores recovered in each split-spoon will be screened in the field for the presence of hydrocarbon constituents through visual examination and using a Photo-ionization Detector (PID). Soil samples will be selected based on staining, odor and elevated PID values and submitted to the laboratory for chemical analysis, according to the SOP entitled "SOP for Sample Target Zone and Sample Selection". These samples will be collected to assist in the identification and quantification of the vertical distribution of selected constituents that may be present in soils.

Borehole cuttings will be both continuously screened for volatile organic compounds (VOCs) and visually examined for signs of staining; those cuttings with either VOCs detected or heavy staining will be stockpiled separately from those soils that are VOC free.

If unsaturated conditions are unexpectedly encountered in boreholes intended for monitoring well completion, the borehole will be left open for 24 hours to determine whether low permeability conditions are retarding groundwater movement. If a groundwater level is observed after the 24-hour period, the level will be noted and the borehole abandoned as described in the SOP entitled "SOP for Boring/Well Abandonment." Boreholes not intended for monitoring well completion will be abandoned as described in the SOP entitled "SOP for Boring/Well Abandonment."

Boreholes intended for monitoring well completion will be completed as described in the SOP entitled "SOP for the Completion of Groundwater Monitoring Well Boreholes."

Upon completion of the drilling, the boring will be surveyed as per the procedures detailed in the SOP entitled "SOP for Surveying Sampling Locations," and abandoned following the procedures detailed in the SOP entitled "SOP for Abandoning Boreholes/Wells."

A-3

SOP FOR THE COMPLETION OF GROUNDWATER MONITORING WELL BOREHOLES

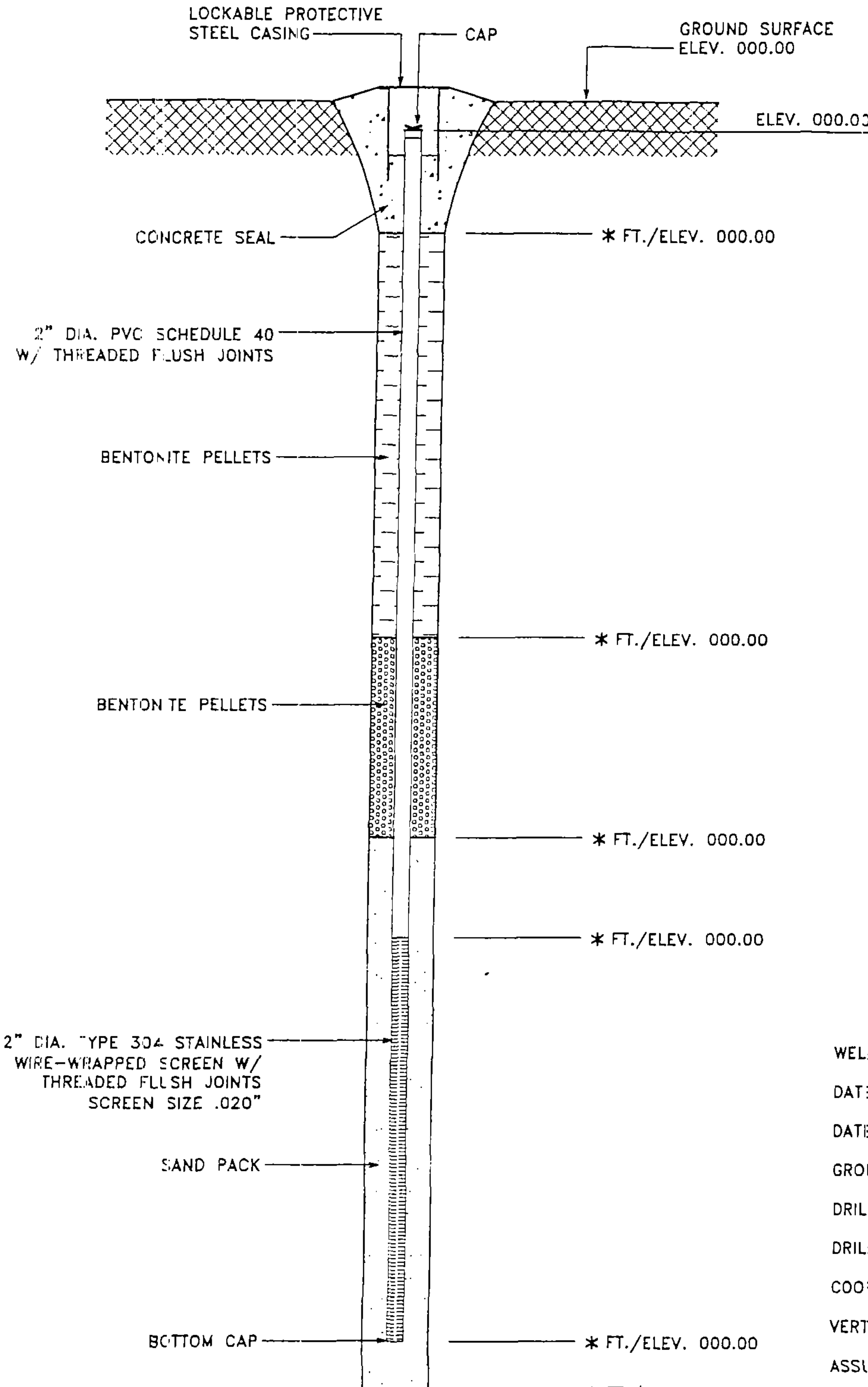
Prior to the drilling of soil borings, scheduled drilling sites will be cleared for utilities and structures by the environmental contractor or their subcontractors. A Utilities and Structures Checklist will also be filled out and signed prior to drilling and excavation activities. A copy of this form is included in Attachment F.

Drilling will be accomplished using either a direct-push rig (Geo-Probe® type), or hollow-stem auger or air/mud rotary or percussion drilling equipment or other applicable drilling rig capable of collecting continuous samples (split-spoon and/or dry core barrel) from the surface to the base of each hole. During drilling of soil borings, a continuous, descriptive, lithologic log will be prepared by a qualified geologist or geotechnical individual based on an examination of the split-spoon samples and soil cuttings. A copy of a blank boring log form is included in Attachment F. In the event that the soil boring can not be completed to a satisfactory depth, an alternative site may be chosen.

Continuous split-spoon samples will be obtained at each soil boring. The soil cores recovered in each split-spoon will be screened in the field for the presence of hydrocarbon constituents through visual examination and using a Photo-ionization Detector (PID). Soil samples will be selected based on staining, odor and elevated PID values and submitted to the laboratory for chemical analysis, according to the SOP entitled "SOP for Sample Target Zone and Sample Selection. These samples will be collected to assist in the identification and quantification of the vertical distribution of selected constituents that may be present in soils.

Borehole cuttings will be both continuously screened for volatile organic compounds (VOCs) and visually examined for signs of staining; those cuttings with either VOCs detected or heavy staining will be stockpiled separately from those soils that are VOC free.

Upon completion of the drilling, the groundwater monitoring well will be constructed according to the procedures detailed in the SOP entitled "SOP for Groundwater Monitoring Well Construction," and surveyed according to the procedures detailed in the SOP entitled "SOP for Surveying Sampling Locations."



WELL NUMBER ____________

DATES DRILLED ____________

DATE INSTALLED ____________

GROUND WATER ELEV.

DRILLER ____________

DRILLING METHOD ____________

COORDINATES ____________

VERTICAL DATUM: ____________

ASSUMED ________ U.S.G.S. ________

* DEPTH BELOW ____________

MONITORING WELL COMPLETION DETAIL - EXAMPLE

SECOR

International Incorporated

PROJECT TITLE
LOCATION
SITE LOCATION

JOB NO. 000.00000.000

FIGURE 0

DRAWING FILE INFO DATE

A-4

SOP FOR COMPLETING FIELD LOGS OF BORINGS

Borings installed on the Site, except those specifically excluded in the RI/FS Work Plan, are to be geologically logged during drilling activities. The following procedures are to be followed for the logging of borings at the Site:

1. As much information as possible is to be shown in the heading of each log. This includes, but is not limited to:
   - Project name and project identification number;
   - Identification of borehole;
   - Name of drilling company and lead driller;
   - Make, model, type, and size of drilling equipment used;
   - Start and end date of drilling
   - Name(s) of field personnel present;
   - Total depth of borehole; and
   - Depth to first encountered water.

2. Each log is to begin with a description of the surface, i.e., native, paved with asphalt, paved with concrete, and such. If any concrete is cut to open the hole, the thickness will be noted.
3. Every foot will be accounted for, with no gaps. If an interval is not sampled, it will be noted. If an attempt is made to sample an interval, but there is no recovery, it will be noted.
4. Complete construction details are to be detailed for each well on a standard well construction form (Attachment F). Construction details should include:
   - A description of the type and length of casing i.e., 20' of 2" inner diameter (id) Schedule 40 polyvinyl chloride (PVC) casing;
   - Length and depths of the top and bottom of the screened interval;
   - Screen slot size;
   - Depths of the top and bottom of the filter pack;
   - Filter pack materials and sand size;
   - Depths and types of bentonite seals;
   - Detail of the use of grout; and
   - Detail of the surface completion (i.e., stick up, flush mounted).

5. The number of bags of sand, bentonite, and grout used will be counted. These numbers will be compared daily with the driller's daily report.

*** The purpose of the field notes and logs is to document observations. They should not be used to state general interpretations (i.e. highly permeable, potential source, ugly).

A-5

SOP FOR SOIL DESCRIPTIONS

Soils logged during Site investigations (i.e. drilling activities) will be described in the following manner:

Soil descriptions will be recorded on a standard Boring Log Form, an example of which is presented in Attachment F. The following categories will be included in Boring Logs, in the listed order:

1. The most current Unified Soil Classification System (USCS) Group Symbol (see page 8).
2. Color (field moisture condition according to Munsell Soil Color Chart or geotechnical gauge).
3. Group Name.
4. Grain Size Range (unless describing a clay).
5. Shape/Angularity of Grains (unless describing a clay).
6. Consistency (SOFT, HARD, LOOSE, etc.), and plasticity for Clays and Silts.
7. Additional Observations (organic material, roots, construction debris, fossils, etc.).
8. Contacts (both sharp and gradational).
9. Moisture Content (DRY, MOIST, WET).
10. Odor with descriptions limited to NO ODOR, SLIGHT ODOR, or STRONG ODOR. No other adjectives to describe an odor will be used.
11. Staining.
12. The total depth of each hole.

In addition to the above items, first encountered groundwater and the static water level are also to be noted on the Boring Log Form.

UNIFIED SOIL CLASSIFICATION SYSTEM

UNIFIED SOIL CLASSIFICATION SYSTEM

IDENTIFICATION PROCEDURES				SYMBOL	TYPICAL NAMES
COARSE GRAINED SOILS	GRAVELS > 50% of coarse fraction is larger than No. 4 sieve	CLEAN GRAVELS	Wide range in grain size and substantial amounts of all intermediate particle sizes	GW	Well-graded gravels, gravel-sand mixtures, little or no fines
			Predominantly one size or a range of sizes with some intermediate sizes missing	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines
		GRAVELS WITH FINES	Non-plastic fines (see ML below for identification procedures)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures
			Plastic fines (see CL below for identification procedures)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures
	SANDS >50% of coarse fraction is smaller than No. 4 sieve	CLEAN SANDS	Wide range in grain size and substantial amounts of all intermediate particle sizes	SW	Well-graded sands, gravelly sands, little or no fines
			Predominantly one size or a range of sizes with some intermediate sizes missing	SP	Poorly graded sands, gravelly sands, little or no fines
		SANDS WITH FINES	Non-plastic fines (see ML below for identification procedures)	SM	Silty sands, poorly graded sand-silt mixtures
			Plastic fines (see CL below for identification procedures)	SC	Clayey sands, poorly graded sand-clay mixtures

		DRY STRENGTH	DILATANCY	TOUGHNESS	FOR FRACTION SMALLER THAN No. 40 SIEVE	
FINE GRAINED SOILS	SILTS AND CLAYS LL<50	None-slight	Quick-slow	None	ML	Inorganic silts and very fine sands, silty or clayey fine sands with slight plasticity, rock flour
		Medium-high	None-very slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
		Slight-medium	Slow	Slight	OL	Organic silts and organic silt-clays of low plasticity
	SILTS AND CLAYS LL>50	Slight-medium	Slow-none	Slight-medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts
		High-very high	None	High	CH	Inorganic clays of high plasticity, fat clays
		Medium-high	None-very slow	Slight-medium	OH	Organic clays of medium to high plasticity
HIGHLY ORGANIC SOILS		Readily identified by color, odor, spongy feel and frequently by fibrous texture			PT	Peat and other highly organic soils

UNIFIED SOIL CLASSIFICATION SYSTEM

UNIFIED SOIL CLASSIFICATION SYSTEM					
IDENTIFICATION PROCEDURES				SYMBOL	TYPICAL NAMES
COARSE GRAINED SOILS	GRAVELS > 50% of coarse fraction is larger than No. 4 sieve	CLEAN GRAVELS	Wide range in grain size and substantial amounts of all intermediate particle sizes	GW	Well-graded gravels, gravel-sand mixtures, little or no fines
			Predominantly one size or a range of sizes with some intermediate sizes missing	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines
		GRAVELS WITH FINES	Non-plastic fines (see ML below for identification procedures)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures
			Plastic fines (see CL below for identification procedures)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures
	SANDS >50% of coarse fraction is smaller than No. 4 sieve	CLEAN SANDS	Wide range in grain size and substantial amounts of all intermediate particle sizes	SW	Well-graded sands, gravelly sands, little or no fines
			Predominantly one size or a range of sizes with some intermediate sizes missing	SP	Poorly graded sands, gravelly sands, little or no fines
		SANDS WITH FINES	Non-plastic fines (see ML below for identification procedures)	SM	Silty sands, poorly graded sand-silt mixtures
			Plastic fines (see CL below for identification procedures)	SC	Clayey sands, poorly graded sand-clay mixtures

		DRY STRENGTH	DILATANCY	TOUGHNESS	FOR FRACTION SMALLER THAN No. 40 SIEVE	
FINE GRAINED SOILS	SILTS AND CLAYS LL<50	None-slight	Quick-slow	None	ML	Inorganic silts and very fine sands, silty or clayey fine sands with slight plasticity, rock flour
		Medium-high	None-very slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
		Slight-medium	Slow	Slight	OL	Organic silts and organic silt-clays of low plasticity
	SILTS AND CLAYS LL>50	Slight-medium	Slow-none	Slight-medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts
		High-very high	None	High	CH	Inorganic clays of high plasticity, fat clays
		Medium-high	None-very slow	Slight-medium	OH	Organic clays of medium to high plasticity
HIGHLY ORGANIC SOILS		Readily identified by color, odor, spongy feel and frequently by fibrous texture			PT	Peat and other highly organic soils

A-6

SOP FOR ROCK DESCRIPTIONS

Rock logged during Site investigations (i.e., drilling activities) will be described in the following manner:

Rock descriptions will be recorded on a standard Boring Log Form, an example of which is presented in Attachment F. The following categories will be included in Boring Logs, in the listed order:

Type of Rock

1. Rock Name (caps - formation name if known).
2. Color according to GSA rock color chart. If rock color chart is not available, use Munsell Soil Color Chart or geotechnical gauge and note so on the log.
3. for sedimentary rock, approximate percentages of fines, sand, and gravel. For example 30% fines, 70% very fine to fine sand.
4. SPACE HOLDER FOR STRATIFICATION.
5. Mineralogy, textural and structural features.

Physical Condition of Rock

6. Nature of the contact; sharp, gradational, or erosional. The log should show a solid line angled across the depth range of a gradational contact. Dashed line for inferred contacts.
7. Nature of fracturing including degree, minimum, maximum, and most common spacing.
8. Further describe fractures including:
    - 8.1 Presence or absence of fracture filling materials
        - 8.1.1 CLEAN - No fracture filling material
        - 8.1.2 STAINED - Coloration of rock only; no recognizable filling material
        - 8.1.3 FILLED - Fractures filled with recognizable material
    - 8.2 Separation of fracture walls
        - 8.2.1 CLOSED - 0
        - 8.2.2 VERY NARROW - 0-0.1 mm
        - 8.2.3 NARROW - 0.1-1.0 mm
        - 8.2.4 WIDE - 1.0-5.0 mm
        - 8.2.5 VERY WIDE - 5.0-25.0 (+) mm
    - 8.3 Fracture roughness classification
        - 8.3.1 SMOOTH - Appears smooth and is essentially smooth to the touch; may be slickensided.
        - 8.3.2 SLIGHTLY ROUGH - Asperities on the surfaces; they are visible and can be felt.
        - 8.3.3 MEDIUM ROUGH - Asperities are clearly visible and surface feels abrasive.
        - 8.3.4 ROUGH - Large angular asperities can be seen.
        - 8.3.5 VERY ROUGH - Near vertical steps and ridges occur on the surface.

Remember that fractures oriented 66-70 degrees to the core axis are suspect compressional/rotational shears induced by the coring process.

9. Hardness, described as follows:
   - 9.1 SOFT - Reserved for plastic material that can be easily molded with fingers.
   - 9.2 FRIABLE - Easily crumbled by finger pressure.
   - 9.3 LOW HARDNESS - Deeply gouged (1/8 inch to 1/4 inch) or carved with pocket knife.
   - 9.4 MODERATE HARDNESS - Readily scratched with knife; scratch leaves heavy trace of dust.
   - 9.5 HARD - Difficult to scratch with knife; scratch produces little powder and is often faintly visible.
   - 9.6 VERY HARD - Cannot be scratched with knife.
10. Weathering with respect to alteration, discoloration, and fracture condition described as follows:
    - 10.1 DEEPLY WEATHERED - Moderate to complete alteration of minerals; discoloration deep and through; all fractures extensively coated.
    - 10.2 MODERATELY WEATHERED - Slight alteration of minerals; discoloration moderate or localized and intense; thin coatings or stains.
    - 10.3 WEAKLY WEATHERED - No alteration of minerals; discoloration slight, intermittent and localized; few stains in fracture surfaces.
    - 10.4 FRESH - Unaltered; no discoloration; none to few stains on fractures.
11. Moisture (DRY, MOIST, or WET)
12. Presence of non-aqueous phase liquid (NAPL)
13. Odor with descriptions limited to NO ODOR, SLIGHT ODOR, or STRONG ODOR. No other adjectives to describe an odor will be used.
14. The total depth for each hole.

In addition to the above items, first encountered groundwater and the static water level are also to be noted on the Boring Log Form

A-7

SOP FOR BORING/WELL ABANDONMENT

Following installation and surveying (by either a professional surveyor or a field team using a global positioning system (GPS), soil borings that will not be converted to groundwater monitoring or extraction wells will be abandoned. In addition, project requirements and/or field conditions may require the occasional abandonment of constructed and/or partially constructed wells. The following minimum requirements for abandoning wells, and soil borings, as required by the Illinois Environmental Protection Agency (IEPA) and based upon previous investigations of the Site geology and hydrology, are presented below:

- All removable casing and or tubing will be removed.
- The hole will be filled, from the total depth to the top of all saturated zones, with cement, bentonite, or a mixture of the two. Expanding cement is preferred in contaminated zones, while bentonite pellets are suggested in uncontaminated, saturated zones. The hole is not to be backfilled with cuttings, regardless of whether they have been characterized as clean or dirty.
- A mixture consisting of cement and 2 to 5 percent bentonite will be used as a surface seal, from the top of all saturated zones to the ground surface.
- A mounded expanding cement collar will be placed at the ground surface in order to divert surface drainage and prevent the intrusion of water into the abandoned hole.
- Borehole seals will be installed using the "tremmie pipe" method to ensure a proper seal.
- A standard abandonment form must be completed, and a State Abandonment Report will be filed with the proper agency.

Note: The above procedures are to be performed by a licensed driller, per applicable State requirements.

A-8

SOP FOR SURFACE SOIL/WASTE SOLIDS SAMPLING

Surface soils and non-soil solids will be sampled throughout the Site at locations specified in the RD Work Plan. Surface soil sampling will be preformed to assess the lateral extent of contamination in surface or near surface soils. Upon collection, surface soils/solids will be field screened for the presence of hydrocarbon constituents through visual examination and using a photo-ionization detector (PID), according to the procedures detailed in Section 3.0 of the FSP, then submitted to the laboratory for chemical analysis. These samples will be collected to assist in the identification and quantification of the vertical distribution of selected hydrocarbon constituents that may be present in soils, and provide information for RD.

The procedures listed below are to be followed for collecting surface soil and non-soil solid samples:

1. Record the sampling location and identification on a standard soil sampling form and the field log book.
2. Collect the sample from the predetermined depth. Samples will be collected using one of the following:
   - Decontaminated soil sampler;
   - Decontaminated stainless steel spoon; or
   - Decontaminated shovel.

3. Upon collection of the sample, immediately field screen each sample as detailed in Section 3.0 of the FSP.
4. Label necessary sample containers with the project number, sampling location, depth, date, time, analysis to be performed, and the initials of the sample personnel prior to or immediately after sampling each interval.
5. Either field-preserve the selected samples, collect them in Encore® samplers, or prepare them according to the sample bottle requirements listed in Table 4-2 of the FSP. Handle and submit the samples as described in the applicable SOP.
5. Describe the lithology of the sampling location according to the procedures detailed in the logging and soil and rock description SOPs.
6. Decontaminate sampling equipment as per the procedures in the applicable SOPs.
7. Dispose of wastes according to the procedures detail in Section 6.0 of the FSP.

A-9

SOP FOR SOIL SAMPLE TARGET ZONE AND SAMPLE SELECTION

The remedial Design (RD) field sampling effort will generate numerous soil samples. Field screening will be conducted on the soil samples in order to identify subsets of samples that require further analysis conducted by a laboratory. This SOP describes the rationale for field screening and selection of samples to be transferred to an analytical laboratory.

The determination of the sampling intervals and types of samples collected will differ depending on the location of the boring. The subsurface investigation can be divided into two groups:

1. Borings within the RCRA outside container storage area (OSA).
2. Borings located outside the OSA.

Determination of Target Sample Zones

Borings located within the OSA

The eight borings that are located within the OSA will require a soil sample submitted to the laboratory for:

- Every two foot interval from the ground surface to the water table (approximately 30 feet below ground surface).
- Sample selection within each two foot interval will be determined by PID screening. PID headspace analysis (samples allowed to equilibrate in a plastic bag for approximately 10 minutes).
- If there are no elevated PID readings the sample shall be collected on the basis of visual staining.
- If no samples in the interval appear to be impacted, one sample must be selected at the discretion of the sampler to send to the laboratory for confirmatory analysis.
- Samples will be analyzed for:
  - VOCs
  - DRO
  - RCRA TCLP metals

Borings located outside of the OSA

The remaining twenty-two boring locations outside of the OSA will require a soil sample sent to the laboratory for:

- Up to two samples collected from the interval between the ground surface and the water table (approximately 30 feet below ground surface).
- Sample selection within the interval will be determined by PID screening. PID headspace analysis (samples allowed to equilibrate in a plastic bag for approximately 10 minutes).

- If there are no elevated PID readings the sample shall be collected on the basis of visual staining.
- If the highest PID reading and staining are in two different intervals then a sample will be collected from the point of the highest PID reading and a sample will be collected from the point of the greatest staining.
- If no samples in the interval appear to be impacted, one sample will be collected at the water table interface (from just above the saturated zone) to send to the laboratory for confirmatory analysis.
- Samples will be analyzed for:
  - VOCs
  - DRO

See the appropriate SOPs for details on: Conducting Field Screening Using a PID (A-16), Subsurface Soil Sampling (A-14), Quality Control Sampling (A-26).

Samples collected for possible analysis but which do not meet the laboratory selection criteria may be disposed of according to the procedures detailed in Section 6.0 of the FSP.

A-10

SOP FOR HYDAC™ TEMPERATURE MEASUREMENT

Summary of Method and Equipment

The temperature probe built into the Hydac™ sample cup will be used to measure groundwater temperature, and will provide the basis for setting the Hydac™ temperature adjustment knobs.

Procedure

The Hydac™ measurement switch should be toggled to the 'Temperature' position. Groundwater will then be decanted from either the disposable bailer or the pump sample port tube into the Hydac™ sample cup, while the Hydac™ read-out switch is depressed. Groundwater will continue to be decanted into the sample cup until the reading stabilizes, in order to minimize the influence of ambient air temperature on the measurement. Following temperature measurement, conductivity and pH measurements will be taken.

A-11

SOP FOR HYDAC™ CONDUCTIVITY MEASUREMENT

Summary of Method and Equipment

The conductivity probe built into the Hydac™ sample cup will be used to measure groundwater conductivity.

Procedure

Prior to pH measurement, the Hydac™ measurement switch is to be toggled to the 'Conductivity' position. The conductivity units selector should be set to the 'x 1000' setting. The conductivity is measured by depressing the Hydac™ read-out switch and waiting for the conductivity measurement to stabilize. Following this reading, the groundwater pH will be recorded as below.

A-12

SOP FOR HYDAC™ pH MEASUREMENT

Summary of Method and Equipment

The pH of a sample is measured electrometrically using both the Hydac™ sample cup and the attached Hydac™ pH electrode probe. Groundwater should be analyzed as soon as possible following temperature and conductivity measurement to avoid changes in pH caused by changes in the chemical equilibrium of the sample.

Calibration Procedure

Prior to each daily use, the pH of the Hydac™ is to be calibrated as follows:

1. Portions of pH 4.0 and 7.0 standards will be placed into clean containers;
2. The attached electrode will be placed into the pH 7.0 solution. After setting the Hydac™ temperature adjustment knob to the approximate temperature of the samples to be screened, the pH will be read and adjusted to read 7.0, using the 'Zero' adjustment knob;
3. The electrode will be removed from the solution and rinsed with distilled water, and then placed into pH 4.0 standard. The pH will again be read and adjusted to read 4.0, with the 'Slope' adjustment knob;
4. The electrode will then be again removed and rinsed with distilled water, and re-inserted into the pH 7.0 standard and adjusted to read 7.0. Steps 2 through 4 will then be repeated until the Hydac™ reads both standard pH solutions to within 0.05 standard units. The final readings, date, and time will then be recorded in the Hydac™ calibration log.

Procedure

Following groundwater temperature and conductivity readings, the attached Hydac™ pH probe is to be inserted into the Hydac™ sample cup, and gently swirled within the groundwater. The Hydac™ read-out button will then be depressed and held down until the pH reading stabilizes. This value will then be recorded.

SOP FOR HYDAC™ METER OPERATION

The Hydac™ meter is a multi-function instrument used to measure the pH, conductivity, and temperature of an aqueous sample. The following procedures will be used to operate the Hydac™ meter during sampling activities:

1. The Hydac™ will be calibrated according to manufacturers' specifications. A minimum of two standards will be used when calibrating for pH (see SOP for Hydac™ pH Measurement);
2. A minimum of two standards and a blank (distilled or deionized water) will be used when calibrating for conductivity;
3. Both the Hydac™ sample cup and the pH probe will be thoroughly decontaminated with an Alconox™ or similar low-phosphate cleaning agent solution, and rinsed with de-ionized water prior to collecting groundwater measurements from each well or sampling station;
4. As outlined in Section 3.0 of the FSP, temperature, conductivity, and pH will be measured immediately following each well purge volume, or following the one-gallon sample port purge in operating groundwater extraction/recovery/production wells. Groundwater will be decanted into the Hydac™ sample cup for measurement;
5. Prior to insertion of the pH probe into the Hydac™ sample cup, temperature and conductivity measurements, in that order, will be recorded. pH will then be recorded;
6. Readings will be recorded to three significant figures for pH and conductivity, and two significant figures for temperature;
7. The Hydac™ sample cup and pH probe will be thoroughly rinsed between readings at each individual well or sampling station; The equipment will be decontaminated, as in Step 3, between wells or sampling stations;
8. The Hydac™ will be re-calibrated prior to each day of use, and again at the end of each day, in order to verify that it kept its calibration throughout the day. Abnormalities will be thoroughly documented and corrected by qualified personnel; and
9. The temperature adjustment knobs on the Hydac™ will be set to the approximate temperature of the samples to be screened prior to initial Hydac™ use, and adjusted throughout the day as necessary.

A-14

SOP FOR FIELD SCREENING USING A PHOTO-IONIZATION DETECTOR

A photo-ionization detector, such as a Rae Systems™ MiniRae™, will be used to field screen soil and non-soil solids for the presence of volatile organic compounds (VOCs). The following procedures are to be followed for the use of the photo-ionization detector (PID), after the initial core-barrel VOC screening described in Section 3.0 of the FSP.

As a first step in PID field screening, immediately reserve two representative portions of each soil sample;

- One portion (for possible Encore® sampling or field preservation and laboratory analysis) should be used to fill containers supplied by the analytical laboratory. Note: the number and type(s) of containers will be location-specific. Those containers should be labeled and stored in a cooler on ice.

- The other portion (for field screening) should be placed into an appropriately sized resealable Ziploc® or equivalent bag. Following bagging, the steps listed below should be followed:

  1. Seal and label the bag with the borehole identification and the depth of the sample.
  2. Transport the bagged soil to the on-Site field laboratory. Allow the bag to equilibrate for approximately ten minutes.
  3. Insert the probe tip of the PID into the bag. Obtain a measurement of total VOCs using the PID.
  4. Ensure the PID has been calibrated according to the procedures in the operation manual. In addition, calibrate the PID anytime there is reason to question the PID readings. Note calibrations in the field logbook and in daily, instrument calibration log (Attachment F).

Calibration instructions for the MiniRae 2000:

- Press Mode and N/- for 3 seconds simultaneously
- Press Y/+ fresh air zero
- Press Y/+ to zero
- Wait
- Press
- Press Y/+ fresh...then continue
- Press N/- span
- Press Y/+ after the screen shows apply gas
- Press Y/+
- Wait
- Press Y/+ to accept calibration
- Press Mode twice
- Ready
- Press Y/+
- Proceed with measuring

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SOP FOR METHOD 5035: FIELD PRESERVATION, COLLECTION, AND HANDLING INSTRUCTIONS FOR VIALS

Method 5035 requires ample preservation in the field at the point of collection. The preservative used for the low concentration soil method (0.5 to200 ug/kg) is sodium bisulfate and the preservative used for the medium/high concentration soil method (>200 ug/kg) is methanol. This field collection and preservation procedure is intended to prevent loss of VOCs during sample transport, handling, and analysis. The holding time for VOC analysis is 14 days.

Materials

- 2 de-ionized water preserved pre-weighed vials for low level analysis with magnetic stir bar.
- 2 sodium bisulfate preserved pre-weighed vials for low level analysis. These vials will also contain a small magnet stir bar.
- 1 methanol preserved pre-weighed vial for medium-high level analysis
- 1 non-preserved 4 oz container for percent total solids determination
- 1 syringe
- 1 Power Handle for collecting samples with syringe

Instruction for Sample Collection

1. The blue plate should be in place on the Power Handle (flanges should be pointing to the round end of the handle). A 5g sample will be collected when the plate is in place.

2. Clip syringe into the Power Handle.

3. Using the Power Handle, push the syringe into the soil to collect 5g sample.

4. Unclip syringe from Power Handle and extrude 5g sample into vial.

5. Repeat process for each additional vial.

6. A single syringe can be used to collect sample aliquots for each of the three vials.

7. Mark each sample container with your sample identification. <u>Do not add any additional labels or tape to the pre-tared vials.</u> Store samples at 4°C. VOCs must be analyzed within 14 days of collection.

8. A fourth container needs to be submitted to the laboratory for percent total solids determination. Fill the container provided to capacity. If extractable organic analyses, i.e., semi-volatiles, PNAs, or pesticides/PCBs will be performed, the fourth container should be a 4-oz. glass jar.

Note: Methanol is a flammable substance. If samples will be shipped to the laboratory via couriers such as UPS or Federal Express, DOT labeling requirements must be met.

To meet DOT labeling requirements, the following statement must be affixed to the package: "This package conforms to the conditions and limitations specified in 49CFR 173.4." The CFR reference is 49 – Transportation, Part 173 – Shippers, General Requirements for Shipments and Packaging, Section 173.4 – Small Quantity Exceptions. In our opinion, the pertinent requirements of this reference are as follows:

- The maximum quantity of material per inner receptacle is limited to thirty (30) mL.
- Each inner receptacle is securely packed in an inside packaging with cushioning and absorbent material. The inside packaging cannot react chemically with the material and needs to be capable of absorbing the entire contents of the receptacle. *(Note: the foam container in which the vials are placed meet these requirements.)*
- The inside packaging is securely packed in a strong outside packaging. *(Note: the cooler meets this requirement.)*
- The gross mass of the completed package does not exceed 64 pounds.

An alternative to field preservation is the use of EnCore samplers (or equivalent) as collection and storage devices. Samples collected in this device must be preserved by the laboratory or analyzed within 48 hours of collection.

Please call us if you have questions concerning these requirements.

SOP FOR SUB-SURFACE SOIL SAMPLING

Continuous split-spoon samples will be obtained at each soil boring. The soil cores recovered in each split-spoon will be screened in the field for the presence of hydrocarbon constituents through visual examination and using a photo-ionization detector (PID), according to the procedures detailed in Section 3.0 of the FSP. Soil samples will be selected based on the SOP entitled "SOP for Sample Target Zone and Sample Selection" and submitted to the laboratory for chemical analysis. These samples will be collected to assist in the identification and quantification of the vertical distribution of selected hydrocarbon constituents that may be present in soils, and provide information for the RI/FS and baseline risk assessment.

The procedures listed below are to be followed for collecting Site sub-surface soil samples:

1. Record the borehole location and identification on a standard soil sampling form and the field log book.
2. Upon recovery of the sampler from the borehole, open and immediately field screen each core as detailed in Section 3.0 of the FSP.
3. Based on the results of the field screening, the SOP entitled "SOP for Sample Target Zone and Sample Selection", collect the appropriate number of bagged and jarred samples. Bag samples are to be placed within new, appropriately sized Zip-Lock™ or equivalent plastic bags. Jar samples are to be placed within appropriately sized (based on the proposed analysis to be run, cross-referenced with the sample bottle requirements listed in Table 4-2 of the FSP) new glass sample jars, and closed tightly.
3. Label necessary sample containers with the project number, borehole number, depth interval, date, time, analysis to be performed, and the initials of the sample personnel prior to or immediately after sampling each interval. (see Section 5.0 of the FSP)
4. Select soil samples to be submitted for laboratory analysis based on the SOP entitled "SOP for Sample Target Zone and Sample Selection". Either field-preserve the selected samples, collect them with Method 5035 syringe samplers, and/or prepare them according to the sample bottle requirements listed in Table 4-2 of the FSP. Handle and submit the samples as described in the applicable SOP.
5. Describe the lithology of the core according to the procedures detailed in the logging and soil and rock description SOPs.
6. Decontaminate sampling equipment as per the procedures in the applicable SOPs.
7. Dispose of wastes according to the procedures detail in 6.0 of the FSP.

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SOP FOR WELL DEVELOPMENT

After completion, each well will be developed by surging and/or bailing a minimum of three well (i.e., borehole) volumes prior to sampling. Appropriate well development will help maximize yields and minimize the turbidity of water obtained during sampling. Well development will terminate when both the groundwater turbidity does not decrease after five casing volumes have been purged, and when pH, electrical conductivity, and temperature have stabilized to within +/- 0.2 standard units, 10%, and 2.0 degrees F, respectively.

Well development activities and measurements will be record on a standard development form. A blank copy of this form can be presented in the attached form..

Groundwater removed during development will be containerized and disposed of as detailed in Section 6.0 of the FSP.

SOP FOR GROUNDWATER SAMPLING FROM OBSERVATION/MONITORING WELLS

The following protocol has been developed to obtain groundwater samples that provide representative chemical quality information. The groundwater sampling procedure will consist of the two steps described below: an initial purging of the well, followed by the collection of samples.

Well Purging

Wells will be purged prior to sampling. Purging will consist of the following steps:

1. Identify the well and record its designation on a both a standard Groundwater Sampling Field Data Sheet (Appendix A-17) and the field log book.
2. Unlock the well and remove the well cap, placing in such a way as to prevent it from coming into contact with any contaminated surfaces.
3. Collect groundwater and non-aqueous phase liquid (NAPL) level measurements as described in the SOP entitle "SOP for Collecting Groundwater Level and NAPL Level Measurements," if this procedure has not already been completed. If NAPL is present and the elevation of the water is to be determined, correct the water level, considering the thickness and the density of the overlaying NAPL. **If NAPL is present, do not sample the well groundwater.** See the SOP entitled "SOP for NAPL Sampling."
   Record applicable information on a standard Groundwater Level Measurement Form and in the field logbook.
4. Compute the volume of water in the well based on the total depth of the well measured, the diameter of the well casing, and the height of the water column in the well.
5. Measure the total depth of the well if required. A comparison of this measured depth with the depth of the well at the time the well was completed will indicate if significant sediment accumulation is occurring in the well.
6. Remove three to five times the volume of standing water in the well, using either a bailer, centrifugal pump, peristaltic pump, or a submersible pump, depending on the depth to water and project specific requirements.

   6.1 In cases where a pump is used, use dedicated or new tubing in each well. If a generator is needed, place it downwind of the well. The submersible pump will be cleaned inside and out according to the "SOP on Decontamination" immediately before placement in the well. Make sure the pump is running before it enters the well, in order to prevent introduction of the remnants of the final distilled water rinse into the well.
   6.2 Position and maintain the intake opening of the pump line or pump impellers just below the water to ensure that the well is properly flushed. If there is a decrease in the well's water level as a result of pumping, the intake line should be lowered as needed. In no case should the pump be placed lower than ten feet below the static water level measured in the well. Pump discharge should be used to limit groundwater drawdown in the well.
   6.3 If the well has been purged or developed recently, the water level (the volume of water in the casing) may not have yet recovered or returned to

its static condition. This does not require a change in the evacuation procedures outlined above. Although the actual column of water in the casing under such conditions is less than normally encountered, the removal of three to five times this volume is normally sufficient to provide samples for analysis that are representative of water from the surrounding formation.

6.4 Following the removal of each casing volume of water from the well, field screen the groundwater for pH, conductivity, and temperature, according to the applicable SOPs.

6.5 The purging will be considered complete when the following qualifications are met:

- A minimum of three casing volumes of groundwater have been removed from the well, and;
- The final two measurements of pH, conductivity, and temperature are within 0.2 standard units, 10%, and 2.0 degrees F, respectively.

7. If the well goes dry prior to the removal of the third casing volume, note this, and the number of gallons removed from the well, on the sampling sheet and in the field log book. Gauge the well groundwater level on appropriate intervals to measure recharge. Upon the well reaching 80 percent recovery of its initially recorded static water level, repeat step 6. If the well again goes dry, repeat step 7. If the well goes dry following three consecutive purges, continue on to step 1, Groundwater sample collection procedures. If the well does not reach 80 percent recharge within 24 hours following the first purge, purge the well dry again and sample the next appearance of water. If there is not enough water to collect a full set of samples, note the well as dry and discontinue sampling efforts for that well. Enter "dry" on the groundwater sampling field sheet and in the field logbook for that well.

8. As noted in Section 3.0 of the FSP, disposable nitrile gloves are to be worn and changed between each well, in order to prevent introduction of external contaminants into the groundwater or groundwater sample, and minimize the chance of cross-contamination between wells. Gloves should also be changed if they become visibly stained with NAPL or contaminated materials.

9. Contain and dispose of purge/development water as specified in Section 6.0 of the FSP.

<u>Groundwater sample collection procedures</u>

Following purging activities, wells are to be sampled using the procedures listed below. Unless directed to do otherwise by the Site-specific work plan, collect water samples using disposable, polyethylene bottom-filling bailers. If the well was purged with a disposable bailer, use the same bailer for sampling.

1. Gauge the well with the interface probe (IP), and determine if the well has reached 80 percent or greater recharge. If the well has not reached 80 percent recharge, gauge the well on appropriate intervals to measure the recharge. Once the well reaches 80 percent recharge, continue on to step 2. If the well does not reach 80 percent recharge within 24-hours, note this and sample the well. If there is not enough water to collect a full set of samples, note the well as dry and discontinue sampling efforts for that well. Enter "dry" on the groundwater sampling field sheet and in the field logbook for that well.

2. Lower the bailer into the well slowly and gently, in order to minimize disturbances to the water table and to avoid aerating the sample.
3. Remove the bailer carefully and gently pour the water sample into the sample containers to minimize the volatilization of organic compounds.
   - Collect duplicate samples directly from the bailer with each sample receiving equal amounts to ensure sample uniformity.
   - If a bailer will not hold the volume of water necessary to immediately fill the sample containers, each container will receive an equal amount from each full bailer.
   - During the sampling of such wells, cap partially filled sample bottles and keep out of sunlight, as delays in obtaining adequate sample volume could otherwise jeopardize the representativeness of the samples.
4. Once the samples have been collected, prepare and preserve them in accordance with recommended USEPA procedures and the Site specific work plan.
5. In general and whenever possible, collect groundwater (as well as surface water, soils, and sediment samples) with the intent to first fill sample containers designated for volatile organic analysis. Follow this by filling containers for semi-volatile organic analyses, metals analyses, and major cation/anion analyses.
6. Upon completion of sampling, cover and lock the well, and remove the sampling materials from around the well.
7. Disposable items, such as bailers, rope, cleaning rags, and gloves, should be disposed of as per the guidelines in Section 6.0 of the FSP.

Well Purging and Groundwater Sampling Equipment

The following field equipment is required for well evacuation and sampling:

Field book, pens, marking pens, and labels.
Kim-wipes, disposable gloves.
NAPL/water level indicator.
Distilled water, sprayer.
Alconox® or equivalent low-phosphate cleaning agent solution.
Disposable polyethylene bailers, or centrifugal, peristaltic, and/or submersible pumps, with appropriate tubing.
Tools for opening wells.
Keys for well locking caps.
Graduated pail and 5-gallon purge buckets.
Coolers and ice.
Hydac ™ meter.
Purge water container (i.e., 200-gallon tank).
Bailer cord.
Chains-of-Custody and field forms.
Sample containers.

SOP FOR GROUNDWATER SAMPLING FROM RECOVERY WELLS

Groundwater recovery/extraction wells that **do not** have dedicated pumps in-place will be sampled according to the procedures detailed in the SOP entitled "SOP for Groundwater Sampling from Observation/Monitoring Wells."

In groundwater recovery/extraction wells with dedicated pumps in-place, two separate groundwater purging and sampling techniques will be utilized. In all cases, the wells will be gauged according to the procedures detailed in the SOP entitled "SOP for Collecting Groundwater Level and NAPL Level Measurements" prior to sampling.

Well Purging

1. In cases when the dedicated groundwater recovery/extraction pump is in operation, it is unnecessary to purge three to five casing volumes from the well. Rather, the pump groundwater discharge manifold valve is to be shut off and the sample port valve is to be opened. Approximately one gallon of groundwater is then to be purged though the well sample port, in order to clear the port and sample hose of any contaminants or debris. Temperature, pH, and conductivity readings are then to be measured and recorded according to the procedures detailed in Section 3.0 of the FSP. The sample is then to be collected according to the **Groundwater Sample Collection Procedures** listed below, from one pump stroke discharge, if possible.

2. In cases where the dedicated pump is not operating, it will be necessary to purge three to five casing volumes of groundwater through the pump discharge line. Calculate the correct purge volumes as per the applicable procedures described in the SOP entitled "SOP FOR GROUNDWATER SAMPLING FROM OBSERVATION/MONITORING WELLS". Groundwater screening and sampling will then follow the procedures listed above in Step 1.

Groundwater Sample Collection Procedures

Samples from recovery/extraction wells are to be decanted into the appropriate sample containers, as detailed in Table 4-2 of the FSP, through the well sample port and any associated dedicated sample tubing. In addition, the procedures listed below are to be followed:

- Collect duplicate samples directly from the bailer with each sample receiving equal amounts to ensure sample uniformity.
- If a single pump stroke will not supply the volume of water necessary to fill all the sample containers, each container will receive an equal amount from each pump stroke.
- During the sampling of such wells, cap partially filled sample bottles and keep out of sunlight, as delays in obtaining adequate sample volume could otherwise jeopardize the representativeness of the samples.
- Once the samples have been collected, prepare them in accordance with recommended United States Environmental Protection Agency (USEPA) procedures and the Site specific work plan.
- In general and whenever possible, collect groundwater with the intent to first fill

sample containers designated for volatile organic analysis. Follow this by filling containers for semi-volatile organic analyses, metals analyses, and major cation/anion analyses.

- Disposable items, such as bailers, rope, cleaning rags, and gloves, should be disposed of as per the guidelines in Section 6.0 of the FSP.

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MANUFACTURERS OPERATING INSTRUCTIONS FOR THE USE OF A THERMO GAS TECH INNOVA-ST (STANDARD MULTI-GAS MONITOR)

Start Up

WARNING

Perform all procedures in a "fresh air" environment (environment known to be free of combustible and toxic gases and of normal oxygen content).

1. Press and hold the **ON/OFF** button for one second. Once **WARMUP COMPLETE** is shown, hold down the **AIR** button for 3 seconds to adjust the Innova to "fresh air" readings ("**demand zero**"). Once **DONE** is shown, the instrument is in the Normal Operation Mode.
2. If applicable, verify that the display reads **0** in the LEL, Toxic 1, and Toxic 2 fields, and **20.9%** in the O_2 field. Any unused channel is blank (if applicable).
3. If applicable, confirm normal operation of the O_2 section. Blow into the probe until the display reaches 19.5%, triggering the alarm.
4. Place the probe into the area to be monitored.

WARNING

Never "demand zero" in a non-fresh air environment.

Operation

In normal operation, your Innova monitors the environment and displays current gas or oxygen concentrations. You can press any button in dimly-lit or dark monitoring area to illuminate the LCD display.

Operator Indications and Suggested Actions

When conditions cause the Innova to reach a preset warn or alarm level, the condition is sensed, and your Innova alerts you with audible and visual indications. Descriptions of common indications, probable (or possible) cause(s), and recommended actions are covered in this section.

CAUTION

Always follow established procedures for an alarm condition. If procedures do not exist, please establish an appropriate plan of action.

Warn Indication

A warn indication occurs when a preset warn level is reached.

Visual/audible indications: The reading of the applicable channel blinks. The red LEDs blink and the buzzer sounds in an even, slow pulsing pattern.

Action: Your Innova resets its alarms when normal gas levels return (if at the default setting **AUTO RESET**), or press **ON/OFF** button momentarily if the alarm latch (**MANUAL RESET**) has been enabled.

ALWAYS investigate the cause of any warn indication.

Alarm Indication

An alarm indication occurs if the gas concentration continues to increase (or decrease) to a preset alarm level.

Visual/audible indications: The reading of the channel in alarm blinks, with the red LEDs and the buzzer sounds at a rapid rate.

Action: Your Innova resets its alarms when normal gas levels return (if at the default setting **AUTO RESET**), or press **ON/OFF** button momentarily if the alarm latch (**MANUAL RESET**) has been enabled.

ALWAYS investigate the cause of any alarm indication.

Fail Indication

A fail indication occurs when a sensor or other circuitry no longer functions normally.

Visual/audible indications: The display for a sensor(s) read XXX. The red LEDs are on, and the buzzer sounds continuously.

Possible causes: A sensor may be bad, missing or have a loose connection. An internal circuit fault may have occurred.

Action: Remove the Innova from the monitoring area. Investigate and determine the cause, refer to the Troubleshooting section of your Operator's Manual for specific instructions.

Low Flow Indication

A low flow indication occurs when normal flow is interrupted. The Innova's pump automatically shuts off.

Visual/audible indications: The words PRESS →TO CLEAR are shown, and alternates with the normal and **PUMP FAILED** screens. An X appears where the spinning icon was. The red LEDs alternate, and the buzzer sounds in a pulsing pattern.

Possible causes: Liquid has been drawn into the probe, or an obstruction is present. An internal circuit fault may have occurred. A sensor may not be properly installed. The hydrophobic filter in the probe may be dirty.

Action: Clear away visible obstructions, then press →(**ON/OFF**) to restart the pump. If the problem remains, troubleshoot the probe, hose, sensor(s) or internal flow system for obstructions.

Low Battery Indication

A low battery indication occurs when the battery voltage drops below the battery alarm threshold.

Visual/audible indications: The words **LOW BATTERY** are shown. The red LEDs are on, and the buzzer sound emits a double pulse every 60 seconds.

Probable cause: The batteries have reached the end of useful life.

Action: You must replace alkaline or recharge or replace NiCd batteries before continuing. Refer to the Maintenance Chapter of your Operator's Manual for specific instructions.

CAUTION

This quick reference card does not adequately replace your operator's manual. Refer to the manual for detailed information, or for other indications not covered on this card, such as TWA, PEAK, and STEL, and all other functions.

Thermo GasTech
1.877.GAS.TECH (USA)
www.thermogastech.com
sales@thermogastech.com
In Canada
1.403.291.4700
www.thermogastech.ca
sales@thermogastech.ca

SOP FOR WATER, SOIL, AND WASTE SOLID SAMPLE HANDLING AND TRANSPORT

The interior of the sampling coolers and exterior of soil and groundwater sample containers will be cleaned with deionized water prior to packing samples for transport to the laboratory. Soil, non-soil solid, and groundwater sample packing will follow the general procedures outlined below:

1. Glass sample containers (i.e. volatile organic analysis (VOA) vials, soil jars) and Encore™ samplers will be placed into bubble-wrap bags following labeling, and sealed;
2. Sample containers will be sealed inside an appropriately sized Zip-lock™ or equivalent baggie;
3. VOA vials will be stored inverted, per United States Environmental Protection Agency (USEPA) regulations;
4. Drain plugs on the sample coolers (if present) will be secured, and packing material added to the coolers to protect the VOA vials;
5. The sample cooler will be lined with a new, sealed plastic bag to prevent any ice melt from leaking out of the cooler;
6. Water, soil, and non-soil solid sample containers will be placed on ice in the sample cooler;
7. The remainder of the sample cooler will be filled with packing material to prevent sample containers from making contact with each other or the sample cooler walls;
8. The cooler inner-liner plastic bag will be sealed with packaging tape;
9. Chain-of-custody forms will be placed in a Zip-lock™ bag (or equivalent) that will be sealed within the sample cooler prior to transport;
10. The cooler will be properly closed and sealed with packaging tape, and;
11. Sample coolers will either be hand delivered to the laboratory by field personnel, or transferred to an appropriate shipping service (ex. FedEx™ or UPS™) for delivery to an out-of-town laboratory.

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SOP FOR DECONTAMINATION PROCEDURES

Reusable field instrumentation and sampling equipment will be decontaminated prior to its first use, and between each well/sampling location in which it is used. Two types of decontamination procedures will be employed, depending on the level of visual or otherwise known contamination to which the instrumentation is exposed. Pre-use decontamination will follow the first decontamination protocol listed below.

Instrumentation and equipment that has no signs of visible non-aqueous phase liquid (NAPL), and which has not come in contact with a known source of NAPL, will be decontaminated in the following manner:

1. The instrumentation and sampling equipment will be thoroughly washed with a mixture comprised of approximately 2-tablespoons of Alconox® (or similar low phosphate cleaning agent) per 1-gallon of de-ionized water. A stiff bristle scrub brush will be used if necessary to provide thorough cleaning.
2. The instrumentation/equipment will be triple-rinsed with unused de-ionized water.

Instrumentation/equipment that either has signs of visible NAPL or has come in contact with a known source of NAPL will be decontaminated in the following manner:

1. The instrumentation/equipment will be thoroughly rinsed with tap water to remove sediment and debris.
2. The instrumentation/equipment will be completely and evenly sprayed with laboratory-grade hexane. ***Proper precautions **must** be utilized when using hexane. Use only in adequately ventilated areas, and do not inhale the vapors. FOLLOW GUIDELINES CONTAINED IN THE HEXANE MSDS.***
3. The instrumentation/equipment will be completely and evenly sprayed with laboratory grade methanol.
4. The instrumentation and sampling equipment will be thoroughly washed with a mixture comprised of approximately 2-tablespoons of Alconox® (or similar low phosphate cleaning agent) per 1-gallon of de-ionized water. A stiff bristle scrub brush will be used if necessary to provide thorough cleaning.
5. The instrumentation/equipment will be triple-rinsed with unused de-ionized water.

The effectiveness of the above decontamination procedures will be demonstrated through the periodic use of equipment blanks. A more detailed discussion of the proposed use of equipment blanks is provided in Section 4.0 of the FSP.

Drill rigs or Geoprobes® used on Site will be thoroughly decontaminated prior to their arrival at the Site and prior to initiation of any drilling activities. The rig and its equipment will be thoroughly examined to ensure that there are no significant fuel, hydraulic fluid, transmission oil, and/or motor oil leaks that could create a condition not previously in existence or exacerbate an existing condition.

Once the rig and its equipment (including split-spoon soil samplers and associated drill rods used to obtain soil samples during the drilling of soil borings or monitoring wells) have been thoroughly cleaned and inspected, subsequent decontamination efforts will focus only on those pieces of equipment which actually come into contact with soils or groundwater. No petroleum hydrocarbon based lubricants will be allowed on the drill stems or associated connections. Both the initial comprehensive cleaning of the rig and subsequent decontamination procedures will be performed using either steam cleaning equipment or high-pressure hot water/detergent wash. In addition, casing centralizers and casing handling equipment, if used, will be cleaned prior to use in the construction of monitoring wells.

Decontamination wash solutions and rinsate will be collected and containerized in 5-gallon buckets, 55-gallon drums, or poly tanks. The collected rinsate will be disposed as described in Section 2.0 of the FSP.

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SOP FOR LNAPL SAMPLE HANDLING AND TRANSPORT

1. To ship volatile organics analysis (VOAs) containing non-aqueous phase liquid (NAPL), the following are needed:
   - **New** paint cans (from a hardware store). One can for each VOA to be shipped will be required.
   - Vermiculite or kitty litter.

2. Place each VOA into a small Zip-Lock™ bag.
3. Use the vermiculite or kitty litter to pack the bagged VOAs into the paint cans. Firmly attach the paint can lids. The key is to have enough absorbent material in the paint can to insulate the VOAs from shocks and to absorb the NAPL if a VOA is damaged. Paint cans are available in various sizes. As mentioned previously, match the number of paint cans to the number of VOAs.
4. Pack the paint cans into a cooler. Use packing material to fill in the space around the cans.
5. Place chain-of-custody into a Zip-Lock™ bag and on top of the cans.
6. At least one label stating: "This Package Conforms to Conditions and Limitations Specified in 49 C.F.R. 173.4" must be attached to the outside of the cooler.
7. ***At least two arrow keys*** pointing towards the top of the cooler must be attached to the outside of the cooler.
8. UPS will accept coolers/packages that are marked as described in steps XI and XII. Federal Express <u>will not</u> accept such packages.
9. NAPL samples <u>must</u> be sent to the appropriate analytical laboratory.

***** The shipping instructions listed above are extremely important. Failure to have the combination of an inner container (the VOA), an outer container (the paint can), absorbent material (the vermiculite/kitty litter), and the label and direction arrows mentioned in steps 6 and 7 could result in government fines of $40,000 per violation.**

ATTACHMENT F TO QAPP
BLANK FIELD FORMS

SECOR Project NO.: 13UN.02072.00.0001
March 31, 2003

ATTACHMENT F
TABLE OF CONTENTS

Date 09/12/00
SOP# ________
Rev # DRAFT

UTILITIES AND STRUCTURES CHECKLIST FORM

Project: ____

Location: ______________________ Date: ______________

Instructions. This checklist has to be completed by a *SECOR* staff member as a safety measure to insure that all underground utility lines, other underground structures as well as above-ground power lines are clearly marked out in the area selected for boring or excavation. **DRILLING OR EXCAVATION WORK MAY NOT PROCEED UNTIL LINES ARE MARKED AND THIS CHECKLIST HAS BEEN COMPLETED.**

Assignment of Responsibility. *SECOR* is responsible for having underground utilities and structures located and marked. Preferably, the utility companies themselves should mark out the lines.

Drilling or Excavation Sites. Attach a map of the property showing the drilling or excavation sites, if sites are widely separated, several map(s) clearly indicating the area(s) checked for underground utilities or underground structures and the location of above-ground power lines.

Utilities and Structures

TYPE	NOT PRESENT	PRESENT	HOW MARKED[1]
Petroleum products line			
Natural gas line			
Steam line			
Water line			
Sewer line			
Storm drain			
Telephone cable			
Electric power line			
Product tank			
Septic tank/drain field			
Other			

[1] Flags, paint on pavement, wooden stakes, etc.

Client Approval ______________ ______________ ______________
(with attached map) **NAME** **COMPANY** **PHONE**

Name and affiliation of person who marked out underground lines or structures.

______________ ______________ ______________
NAME **COMPANY** **PHONE**

SECOR International Incorporated *(SECOR)*

Field Team Leader ______________________ Date Completed ______________

P:/conoco/admin/manual/sops/utilstruc.doc

SECOR BORING/MONITORING WELL LOG	DATE	SECOR PROJECT NUMBER	MONITORING WELL/ BORING NUMBER	PA
	WEATHER CONDITIONS	CLIENT	SITE LOCATION	

☐ SOIL BORING ONLY ☐ SOIL BORING COMPLETED AS MONITORING WELL	DRILLING METHOD	SOIL SAMPLING METHOD	LOCATION SKETCH
DRILLING COMPANY	DRILLING START	DRILLING FINSIH	DEPTH TO WATER DURING DRILLING
BORING BACKFILL MATERIAL	BORING LOGGED BY		DEPTH TO WATER AFTER WELL SET

DEPTH (in feet)	BLOW COUNTS Per 0.5 Feet	RECOVERY (Feet)	MOISTURE CONTENT	PENETROMETER READING (TSF)	PID/FID (ppmv)	PROFILE	DESCRIPTION	WELL DIAGRAM

CHECKED BY:	DATE:	▽ INITIAL WATER LEVEL	▼ STATIC WATER LEVEL

AIR MONITORING EQUIPMENT CALIBRATION/CHECK LOG

DATE	INSTRUMENT/ MODEL NO.	SERIAL NO.	BATTERY CHECK OK?	ZERO ADJUST OK?	CALIBRATION GAS (PPM)	READING (PPM)	LEAK CHECK	PERFORMED BY	COMMENTS

AIR MONITORING LOG

DATE	TIME	LOCATION	SOURCE/AREA/ BREATHING ZONE	INSTRUMENT	CONCENTRATION/UNITS	SAMPLED BY

* Submit copies of logs to Director of Industrial Hygiene & Health and Safety, Philip A. Platcow, CIH within 24 hours, if a PEL is exceeded, or personal protective equipment level is upgraded at (617) 232 7355 or via email at pplatcow@secor.com

SECOR *International Incorporated*
GROUNDWATER SAMPLING FIELD DATA SHEET

SECOR PN: ______________________ DATE: ____________ WELL #: ______

FACILITY NAME: ______________________ TEMPERATURE: ________ °F or °C

FIELD PERSONNEL: ______________________ WEATHER: ______________________

FIELD MEASUREMENTS:

A. Static Water Level (SWL) below top of casing/piezometer: ____________ FT. or IN.
B. Thickness of Free Product, if present: ________ Inches ____________ FT. or IN.
C. Total Depth of well (TD) from top of casing/piezomenter: ____________ FT. or IN.
D. Height of Water Column in casing (h=TD-SWL): ____________ FT. or IN.
E. **Useful approximate Purge Volumes (PV) per foot of water for common casing sizes:** ____________ ____________

PURGING METHOD: ______________________ DURATION: ______________________
OBSERVATIONS:

	Time	Turbidity	Color	Sheen	pH	Temp.	Conduct	SWL
1st Volume:								
2nd Volume:								
3rd Volume:								
4th Volume:								
Add'al. Volumes:								

TOTAL VOLUME OF WATER PURGED FROM WELL: ______________________

PURGE WATER STORED / DISPOSED OF WHERE / HOW: ______________________

SAMPLES COLLECTED: Depth to Water at time of sample collection: ______________________

Sample Numbers	Type	Time	Size/Number of Container(s)	Preservative

COMMENTS:

____

____

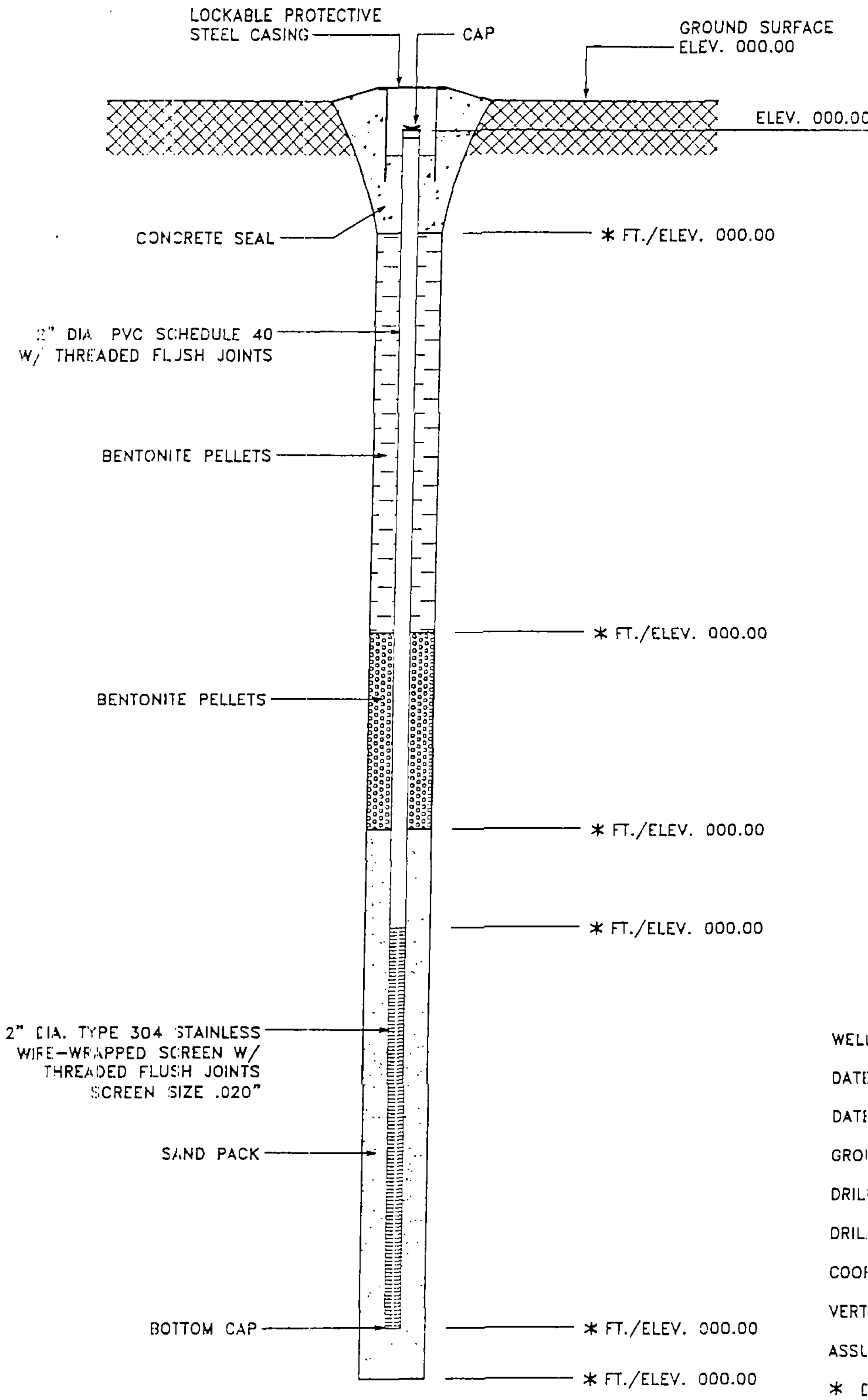
____

Casing Capacities:
2-inch hole .....0.16 gal/lin ft.
4-inch hole .....0.65 gal/lin ft.
6.5-inch hole .....1.70 gal/lin ft.
8-inch hole .....2.60 gal/lin ft.
10-inch hole .....4.10 gal/lin ft.

Recharge Calculation at Time of Sample Collection

Total Depth of Well: ____________
Original Water Column: ________ X 0.80 = - (________)
Collect sample when Depth to Water measures
Less than or equal to: ____________

Signature: ______________________



WELL NUMBER ____________

DATES DRILLED ____________

DATE INSTALLED ____________

GROUND WATER ELEV.

DRILLER ____________

DRILLING METHOD ____________

COORDINATES ____________

VERTICAL DATUM: ____________

ASSUMED ________ U.S.G.S. ________

* DEPTH BELOW ____________

MONITORING WELL COMPLETION DETAIL - EXAMPLE

SECOR
International Incorporated

PROJECT TITLE
LOCATION
SITE LOCATION

JOB NO. 000.00000.000

FIGURE 0